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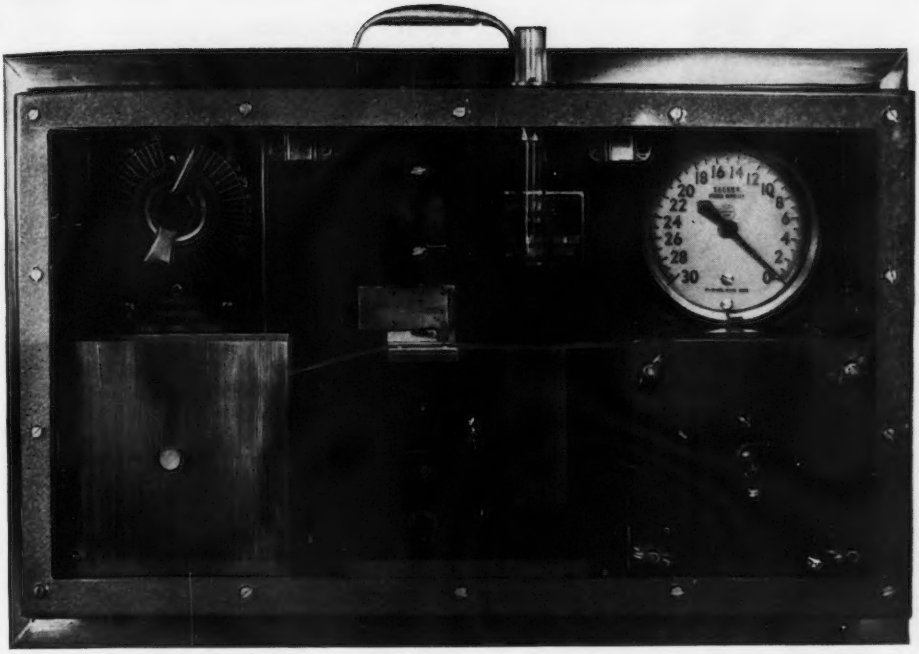

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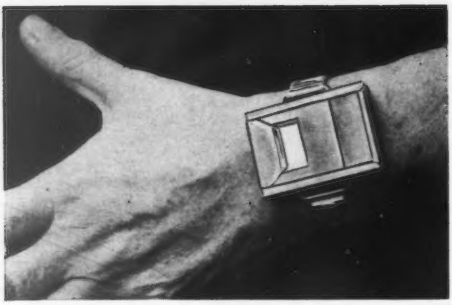

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Critique of Present Efforts to Develop a Damage Risk Criterion*

RAYMOND CARHART, Ph.D

Department of Otolaryngology, and School of Speech, Northwestern University, Evanston, Illinois

A FUNDAMENTAL GOAL of industrial hygiene is to protect workers from noise-induced hearing loss. To this end, we strive to quiet industrial equipment through engineering procedures, to make work areas less noisy through architectural acoustics, and to guard workers through mandatory systems of ear protection. Concomitantly, we conduct audiometric programs to discover incipient hearing impairments in the industrial population and to isolate the noise-susceptible individual.

These several facets of hearing conservation require that the boundaries of noxious noise be discovered, that the conditions which lead to permanent acoustic insult be specified, and that insightful criteria of damage risk be formulated. The contemporary view, as expressed by Harris, is, "A damage-risk criterion specifies the maximum sound pressure level of a noise, as a function of frequency, to which people should be exposed if risk of hearing loss is to be avoided. This criterion includes a specification of the time of exposure, amount of hearing loss considered significant, and percentage of the population to be protected."[1]

The task of implementing this definition has proved a difficult one, yet it must be accomplished. Only to the degree that realistic criteria of safe noise levels are known can adequate steps in protection of hearing be assured without excessive cost and inconvenience to industry.

The complexity of the problem of specifying damage risk criteria is illustrated by the history of efforts to do so. The first criteria, which were based largely on casual observations of industrial and military conditions of noise exposure, were expressed as overall noise levels. Bunch,[2] for example, designated 90 db as the boundary of noxiousness without clarifying the scale of intensity he had in mind. Kryter,[3] by contrast, formulated a very precise boundary of noxiousness when he suggested that extended exposure to sound is safe provided the sound pressure level (SPL) per critical band does not exceed 86 db re 0.0002 microbar. Kryter did not phrase this criterion in terms of sound pressure level per octave band but popular contemporary criteria expressed in this way have evolved from Kryter's premise.[4, 5, 6] These criteria have the merit of being simple and of allowing immediate evaluation of broad-band industrial noise, albeit they may be unrealistic to the extent they retain commitment to the philosophy underlying the critical band concept. This concept, in view of the peculiarities of binaural masking which Hirsh[7] has reported, cannot be accepted as an uncontaminated description of energy relations within the inner ear.

An entirely different approach to the problem was made by McGrath,[8] who combined an analysis of theoretical considerations with observation on industrial workers. He evolved a full-fledged system for designating acoustic noxiousness. McGrath emphasized four points. First, he stressed that the spectral peculiarity of the noise is one determinant of its threat to hearing. Second, McGrath concluded that noxiousness increases systematically as the frequency is raised when the intensity is expressed as sound pressure level per octave band. Third, he demonstrated that noxiousness is graded from conditions of full safety to conditions which are highly destructive. Finally, McGrath proposed a method for scaling complex noises so that highly dissimilar acoustic patterns could be compared.

Hardy[9] developed an alternative system for designating acoustic noxiousness. His system bears some resemblance to McGrath's, but it is based on a set of "equal sensation curves". Hardy derived these curves from available evidence on the frequency sensitivity and the loudness perception of normal hearers. His system has the merit of furnishing a unique weighting of octave bands and of stressing that there is continuous gradation of noxiousness once the boundary of safety is exceeded. Hardy's system is distinctive in the definitive way in which it predicts that broad-band industrial noises should produce the typical noise-induced hearing loss with its maximum at 4000 cps. This and other considerations lead to confidence in Hardy's criterion. It ap-

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, April 30, 1959.

pears to some of us to be a promising approach to the task of specifying the noxiousness of broad-band, sustained industrial noise. True, the details of the criterion may need refinement, but the principles it embodies appear worthy of preservation.

Obviously, if a damage risk criterion is to be put to practical use in industry, the criterion must be expressed as one or two clearly defined boundaries which, when exceeded, become the impetus to appropriate protective action. Hence, the concept of graded noxiousness, which is central in McGrath's and Hardy's systems (and which is scientifically important) can not be preserved in a workaday criterion. True, either system can supply a sharp boundary, and each man has indicated the appropriate lower limit of noxiousness as his system defines it. However, the simplest system for specifying a single boundary as the damage risk criterion for lifetime exposure to broad-band industrial noise is the well-known extension of Kryter's premise developed by Rosenblith *et al.*[4] A more inclusive alternative is to specify the boundary of negligible risk, above which protective procedures are judged desirable, and the boundary of likely damage, above which these measures are made mandatory. The Air Force has done the latter.[10] In any event, the point to remember is that arbitrary compromises are incorporated in any damage risk criterion, so that one consideration in evaluating a criterion is whether the most reasonable compromises have been made.

A particularly important set of compromises, which have greatly extended the range of applicability of damage risk criteria, have evolved from the extensive work of the Z24-X-2 Committee[11], of the Sub-Committee on Noise in Industry[12] and of the Armed Forces National Research Council Committee on Hearing and Bio-Acoustics (CHABA).[13] The analysis of the audiological problems in both military and industrial situations made by these groups has contributed to full realization that damage risk criteria must allow for the diversity of noise exposure which characterizes everyday situations. Adequate criteria must recognize the variations, from one job to another, in the duration of daily exposure to high level noise, as well as the diversity of temporal and spectral characteristics of noise environments. The end result, as Rosenblith has expressed the matter, is that "... at present, we base our criteria on the concept of *noise exposure* in contradistinction to the previously emphasized *noise level*".[14]

Air Force Regulation 160-3[10] illustrates the type of compromise which allows damage risk criteria to incorporate these factors. Four premises serve as the foundation for the regulation. These premises are not new, but they are combined to supply a unique type of criterion.

The first premise is that an allowable lifetime exposure to broad-band noise may be specified. This exposure is defined in the Regulation as the maximum level in which a man may spend twenty-five years during normal work-hours (40 per week), with ears unprotected, without serious danger of damage to hearing. The Regulation accepts a sound pressure level of 85 db per octave band as the presumed limit for full safety.

The second premise is that allowance must be made for the extra noxiousness of unusual spectral features in continuous noise, such as strong pure tone elements and narrow band components.

The third premise is that the total amount of energy received during the day is the final determinant of noxiousness. Therefore, one may compute equivalent exposure times for short imersions in higher noise levels than are allowable for the full work day, and one may specify the limiting exposure time which is not to be exceeded if the total energy to which the worker is exposed is to be maintained below the danger point, as designated by the concept of allowable lifetime exposure.

The fourth premise is that two boundaries, or criteria, are warranted: one at which protection of hearing becomes desirable and a second at which protection is mandatory. The boundary for optional protection is reached when the equivalent exposure time exceeds the allowable lifetime exposure in the strongest octave band between 300 and 4800 cps, while the boundary for mandatory protection is reached when the equivalent lifetime exposure would be 95 db for at least one of these octave bands.

Air Force Regulation 160-3 epitomizes one contemporary philosophy; namely, that a simplified damage risk criterion may properly be employed to determine when to initiate specified protective action.

There is a second major viewpoint, which has superficial similarities to the first one but which actually avoids designating a damage risk criterion. Instead, this second approach indicates sound pressure levels at which management should consider initiating a hearing conservation program for the purpose of assuring that workers are not suffering auditory damage. This philosophy has been well expressed by the Subcommittee on Noise in Industry as follows:

"At the present time our knowledge of the relations of noise-exposure to hearing loss is

much too limited for us to propose *safe* amounts of noise-exposure. We can, however, point to certain noise levels that indicate when it is advisable to initiate hearing conservation programs. These levels will not be general because a different level is needed for different types of noise and different schedules of exposure."[12]

In a very real sense, the foregoing viewpoint represents the present terminus in audiology's search for damage risk criteria. It is the terminus because, as the quotation above reminds us, there are so many gaps in contemporary knowledge. However, a fundamental socio-philosophical problem is equally responsible for our current uncertainty.

The economic and moral goals to be achieved by a hearing conservation program must be clearly stated before the task of selecting damage risk criteria can be undertaken meaningfully. In other words, the major need at present is for full clarification of the social definition of auditory damage. Both the present attitudes toward damage risk criteria and the criteria themselves imply philosophies of social responsibility which are personally accepted by the individuals whose judgment they embody. There is no assurance that these philosophies are generally accepted when applied to the task of defining industry's responsibility for noise-induced hearing loss. For one thing, statistical considerations become important. We are well aware that auditory damage to a particular condition of exposure will vary within the industrial population both in amount and in rapidity of onset. Even though the details of the distribution can not yet be described precisely and even though its characteristics may differ with changes in exposure,[15] some persons suffer more harm to hearing than others and a continuum of susceptibility exists. It becomes an economic impracticality to establish a cut-off point of allowable noise exposure which will offer full protection for every worker. Remember, for example, Rosenwinkel and Stewart[16] found change in acuity at 4000 cps to result from as little as five years of exposure to sound at 80 db SPL per octave band. Consequently, we are faced with the unavoidable fact that any reasonable designation of a damage risk criterion embodies the decision that some of the hearing of some workers is expendable. The problem of selecting the criterion thus becomes one of balancing two factors: namely, (1) the costs to the individual against the cost to society, and (2) the rights of the individual against the rights of society.

The premise that it is necessary to accept a calculated risk for a few workers rather than to assure full protection for every worker appears inhuman until one recalls that this premise is the accepted basis for conduct in most areas of modern life. Our culture accepts the calculated risk of several hundred deaths on the highway rather than the restriction of eliminating all automobile travel over the Labor Day weekend. We estimate in advance the casualties to be expected in a major construction job, and then proceed with the construction. Thus, when we consider the question of hearing loss, the problem becomes one of establishing (through social definition) the value judgment as to what fraction of auditory functions is expendable.

The foregoing philosophy is frequently denied as untenable in our culture. Rosenblith, in discussing the problem of picking a statistical boundary for the damage risk criterion, states, "The representative of a state government who testified before the Z24-X-2 committee reminded us gently that such a position is politically impossible in a democracy, where one has to aim at protecting everyone."[14] Note the interesting fact that the goal is *not to insure* protection for everyone, but "*to aim* at protecting everyone". In this phrasing, there is the tacit assumption that some failure is unavoidable. Of course, as Rosenblith has pointed out, insightful programs of hearing conservation combine criteria for safety with practices of preventive medicine so as to protect almost everyone, provided we define protection realistically in the light of expendable auditory capacities.

Obviously, professional people must contribute importantly to the value judgments which define the limits of auditory expendability. Their specialized knowledge is an essential ingredient to equitable social decision. We must therefore remember that "...informed scientific opinion has come to feel that as long as man's hearing for everyday speech, under everyday conditions, remains protected, we are successfully preventing noise-induced hearing losses from becoming a serious handicap."[14]

The foregoing generalization has been translated into legal terms in various ways. It is embodied, for example, in the regulations which have been adopted in Wisconsin[17] to govern compensation for hearing loss. Here the compensation schedule is restricted to consideration of pure tone thresholds in the 500–2000 cps band. We all recognize that this band represents a very limited definition of the speech range, but it does realistically delimit the segment of the frequency

range which is acoustically critical for understanding ordinary conversational speech.

Stated differently, industrial audiology is in a paradoxical situation as regards contemporary damage risk criteria because these criteria have not been unequivocally related to ultimate social requirements in hearing conservation. Contemporary criteria imply that hearing is to be preserved throughout a very broad range of frequencies.

More specifically, the systems favored by McGrath,[8] by Hardy,[9] and by the Beranek group[6] encompass the range from approximately 20 to 10,000 cps, while the Air Force criterion[10] is based on the sound pressure levels which occur in the four octave bands between 300 and 4800 cps. The paradox becomes clear when one recalls that hearing impairment occurs primarily for frequencies above those of the damaging components in the noise spectrum.[3] The upward spread may be extensive, but the influence on hearing for frequencies lower than the frequencies of the noxious spectral components is limited. Thus, since the recognized contemporary criteria for damage risk require that frequencies above 2400 be taken into account, they place emphasis upon segments of the acoustic spectrum whose noxious effects will be primarily above 2000 cps. Hence, they give value to segments of the spectrum that lie above the critical speech range as defined by such regulations as the Wisconsin schedule of compensation for hearing loss and by most listening demands in everyday life.

Please do not misunderstand. The foregoing comment is not an argument that components above 2400 cps should be ignored. The decision on this point will be implied by the laws and regulations which are adopted throughout the world to govern compensation for hearing losses. Obviously, the outcome may vary from one state or country to another.

The point being stressed is that damage risk criteria should conform to a social definition, which only laws and regulations furnish, as to the range of hearing that is judged critical for protection. It is perfectly possible for research findings to demonstrate, as Rosenwinkel and Stewart[16] have done, that

> "Permanent changes in hearing sensitivity to pure tones occur in the region beyond 2000 cycles per second when the sound pressure level in the environment is steady at 80 db per octave band and exposure is continuous for 10 years and over."[18]

without implying that the damage risk criterion should be established at this level. The major need at present is for full clarification of the definition of critical auditory damage which is to apply to the industrial world. We have drifted into those definitions which are currently in use. We will continue to drift unless the issues are faced squarely and unless positive decisions are made as to what degree of hearing is to be considered indispensable.

Once we are armed with such a definition, the appropriate damage risk criterion can be specified almost automatically. The hearing capacity which is designated as indispensable must be preserved if at all possible, while the hearing capacity which lies outside this range may be considered a luxury whose preservation is optional. As soon as the range is designated, the task becomes one of turning to research findings for the relationships between noise exposure and hearing loss. The proper damage risk criterion can emerge unequivocably from adequate knowledge of these relationships.

Unfortunately, as we are all aware, contemporary understanding of the relationships between noise exposure and acoustic insult is woefully incomplete. Therefore, full refinement of damage risk criteria must await extensive research. On this topic, fortunately, much investigation is in progress, and important facts are emerging continuously. A few major trends which are becoming apparent must be mentioned because of their implications in connection with damage risk criteria.

For one thing, both military experience and recent surveys of industrial populations demonstrate that the human ear is more resistive to excess damage than was thought to be the case when Bunch[2] and his contemporaries gave the first quantitative description of "acoustic trauma". The well-known Z24-X-2 investigation,[11] for example, has contributed importantly to the emergence of this realization. Data obtained in the Z24-X-2 study demonstrate that net hearing losses after years of exposure to substantial industrial noises are often small when allowance is made for presbycusic deterioration. Thus, while the noxiousness of noise is not to be belittled, we can approach the tasks of industrial audiology with a helpful optimism as to the ruggedness of the human auditory system when it is given reasonable protection. Moreover, we can approach the task of establishing the relationship between noise exposure and auditory impairment more realistically from a frame of reference which does not penalize industrial noise for the aging of the worker. Similar realism can be brought to bear on the development of equitable schedules of compensation for hearing disability, which, in turn, must contribute to the evolution of insightful damage risk criteria.

Secondly, understanding of the noise conditions

experienced by workers is being greatly improved through the recent development of measuring cumulations of exposure to fluctuating noise.[19, 20] Cox, for example, describes use of a noise cumulator to study flight deck conditions on an aircraft carrier. The device samples and integrates intensity level as a function of time in such a way that the total exposure which reaches or exceeds each of seven levels is ascertained. Cox states, "One can distinguish the variations in noise exposure among different jobs, the variations in noise exposure with different noise sources, and the variations in noise exposure in different frequency bands."[20]

Now that the methodology for measuring cumulative exposure is available, the task is to conduct the copious research which is required to determine the dependence of hearing loss on cumulative exposure to fluctuating industrial noise.

Simultaneously, we must clarify the conditions under which the concept of equivalent exposure time ceases to be applicable. There must be limits to the brevity and intensity of sound to which this principle applies. Sudden traumatic injury to the ear will occur when these limits are exceeded.[21] Moreover, cumulation of insult may be different when such boundaries are exceeded so that there may be still other kinds of damage to the ear. Therefore, we must have at least two sets of criteria for noxiousness, one marking the boundary of gradual erosion of acuity and the second marking the boundaries for sudden damage.

Thus, a positive research attack on the problem of impact noises and explosively transient sounds is essential if damage risk criteria are truly to be clarified. The great noxiousness of impulsive stimuli has long been recognized, but such sounds have defied incisive classification as destroyers of auditory capacity. The time has passed when industrial audiology can be content to codify the risk criteria for sustained noises and to express industrial and military environments in terms of the simple formula of equivalent exposure time. The importance of transient acoustic features, narrow band characteristics, and cumulative effects other than simple summation must be discovered and incorporated in damage risk criteria.

In the third place, since the human being who is being exposed to noise is the critical factor in the whole picture, research on basic phenomena of audition as these relate to noise-induced hearing loss must be continued.[22] For one thing, we are still highly uncertain as to the relations among the various types of threshold shift which noise produces. At least six phenomena of altered acuity can be observed. Some of these are attributable to adaptation. Others appear to be manifestations of fatigue, but slowly reversing losses and permanent impairment must be considered to be products of physical damage. The complexity of the situation is illustrated by Kopra's statement regarding adaptation and fatigue. He says, "... one must differentiate between (1) the fleeting desensitization of momentary adaptation and the more extended desensitization of equilibrated adaptation, (2) between momentary adaptation and momentary fatigue, (3) between momentary fatigue and prolonged fatigue, and (4) between equilibrated adaptation and prolonged fatigue."[23] Moreover, several of these phenomena evidence progressive effects when evoked repeatedly. It is little wonder that we have failed to discover clear relations between any of these temporary threshold shifts and permanent damage to hearing. One can understand why Summerfield, Glorig and Wheeler[15] have come to the conclusion that the search for tests of noise-susceptibility has not yielded a definitive way of identifying the susceptible person.

A further complication in this regard is the probability that noise-susceptibility is not a state which remains stable throughout life. It may be that an individual's proneness to permanent damage from industrial noise varies radically with his physical condition.[22] Sub-clinical states of endolymphatic hydrops, for example, may produce temporary proneness to acoustic insult. We have observed this phenomenon once in our own laboratory work on noise-susceptibility. Along the same line, Derlacki reports that clinical symptoms of hydrops can appear for the first time following noise exposure.[24] One suspects that in such instances pre-clinical pathology may have been incited to clinical magnitude by the noise exposure, which might well have been innocuous if the pre-clinical state had been absent.

Insofar as the establishment of damage risk criteria is concerned, the foregoing possibility has three important implications. For one thing, research directed toward discovering the minimal conditions of noise which induce hearing loss becomes much more difficult. The principle of safe lifetime exposure is not applicable to the person who is temporarily sensitized to acoustic insult. Secondly, it is problematic from the practical point of view whether damage risk criteria can reasonably be established at the relatively low sound pressure levels which would fully protect the person who is temporarily noise-susceptible.

Finally, there is the moral and legal question as to whether industry should be held accountable for hearing loss resulting from susceptibility caused by physiological states which are independent of working conditions or job demands. The answer to this question will determine

whether it is necessary to establish criteria so low that they forbid a substantial range of exposure levels which would be safe except during temporary noise-susceptibility.

In conclusion, Glorig expressed the limitations which we presently face in attempting to refine damage risk when he wrote, "Setting damage risk levels for noise-exposure presumes a great deal of knowledge of many related factors. . . . We are not prepared to explain susceptibility nor to predict who is more highly susceptible; therefore, we cannot expect to establish criteria for preventing hearing loss in anyone and everyone."[25] Despite this fact, contemporary criteria are sometimes phrased and sometimes employed as though they marked firm boundaries of full protection for the industrial worker. The proper approach is to view contemporary criteria as means of identifying those situations where programs of hearing conservation should be initiated to achieve protection. Improved damage risk criteria will ultimately emerge from the knowledge which research is accumulating and from legal clarification of the socio-economic standards to be satisfied in guarding the hearing of the industrial worker. Only time will tell whether these improved criteria can properly be applied rigidly or whether they must merely remain guides to the need for hearing conservation programs.

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Special Sound Absorptive Materials in Noise Control*

BENJAMIN C. DUGGAR

Engineering Department, E. I. du Pont de Nemours & Co., Wilmington, Delaware

Introduction

RECENTLY, industrial hygiene engineers at du Pont have had occasion to employ special sound absorptive materials in noise control devices. The designation "special" is used to infer that these materials are not normally regarded as acoustical materials. The absence of acoustical properties data for these materials necessitated experimental determination of absorption coefficients and acoustical impedance. It is the purpose of this paper to describe two examples where special sound absorptive materials were required and to discuss the evaluation of their acoustical properties. A summary of the acoustical characteristics of several synthetic fibers and plastic foams is included. Although the examples cited are specific to the chemical industry, the principles illustrated should have wide application.

Background

The effectiveness with which noise control devices function over an indefinite time period depends on many factors. The devices should be unaffected by a wide variety of environmental conditions, require little maintenance and offer a minimum of inconvenience to operating personnel. Numerous examples of effective noise reduction measures for both common and unusual types of noise problems have been described in the literature. Most of the control devices rely to some extent on sound absorptive materials for their effectiveness. However, little information has been published regarding the ability of these materials to maintain their sound absorptive characteristics during continued exposure to corrosive gases, saturated steam, entrained moisture, or mechanical abuse. In addition to acoustical effectiveness, certain applications require that the absorptive material not sift or shed particles which would contaminate a product or dust-free area. If the material must be handled frequently it should not produce dermatitis. These considerations may make it desirable to use a sound absorptive media which has the specifically required physical characteristics, even though not generally recognized as an acoustical material.

Acoustical Characteristics

ACOUSTIC IMPEDANCE

The industrial hygiene engineer engaged in developing noise control devices will often find that acoustical impedance data as well as absorption coefficients are necessary for optimum design. Briefly stated, acoustic impedance refers to the complex ratio of sound pressure to particle velocity. A material with an impedance differing greatly from air will reflect airborne sound, while a material with a similar impedance will not. The resistive and reactive components determine the energy dissipation (sound absorption) and can be related to physical properties of the material. To achieve maximum sound absorption at any given frequency perforated facings may be used to minimize the reactive impedance of an absorptive material while adjusting the resistive component to an optimum value. Bolt[1] has published design charts from which the reactance of perforated facings may be readily calculated. The change in resistive impedance due to addition of a perforated facing may be estimated by use of a chart published by Ingard.[2] However, the absorption coefficients of the material with facing cannot be calculated unless the initial impedance data are available. Lack of published impedance data for common acoustical materials has restricted the use of these techniques employing perforated facings. Although a series of measurements of acoustic impedance of commercial acoustical materials was published in 1940,[3] there is a need for additional data for the many new materials. Manufacturers of acoustical materials can contribute by including such data for their products in addition to the reverberation coefficient presently published in their sales brochures.

Normal absorption coefficients and specific normal acoustic impedance can be rapidly measured by use of the "tube" method. This is described in the tentative ASTM method C384–

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56T. We have used a four-inch diameter tube for several years to derive the optimum combination of the parameters controlling sound absorption by the materials of interest to our noise control program.

MEASUREMENTS FOR SPECIAL MATERIALS

To assist others who have applications requiring use of special sound absorptive materials, we have summarized the results of some of our investigations in the accompanying graphs (Figures 1–6). In these graphs normal absorption coefficients and resistive and reactive impedance have been plotted as a function of frequency.

Synthetic Fibers: The type of fiber and fiber size or denier appear to have less importance than do the thickness and packed density in determining the acoustic characteristics of the fibers we have investigated. Measurements for nylon, Dacron (du Pont polyester fiber), and Orlon (du Pont acrylic fiber) packed to similar densities do not show significant differences in absorption. Typical curves for a batt two-inches thick and packed to 4.5 lb per cubic feet appear in Figure 1. The effects of varying thickness of the fiber batt are shown in Figure 2. Note that although the resistive impedance is similar for the two thicknesses, the reactive component is very thickness dependent. The location of the cut-off frequency below which the normal absorption coefficient falls off rapidly is inversely related to the thickness of the sample if all other

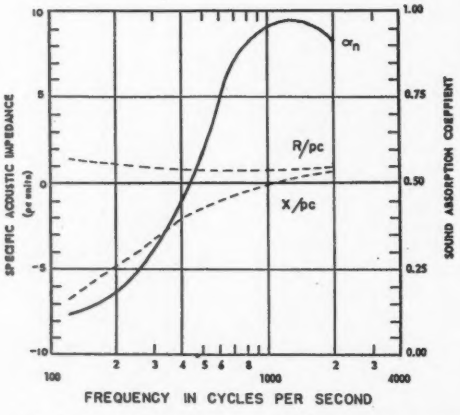

FIGURE 1. Normal absorption coefficients and specific acoustic impedance for a typical batt of synthetic fibers, 2 inches thick and packed to 4.5 lb per ft³. The sample was faced with a perforated plastic plate, $\frac{3}{32}$ in. thick, with $\frac{3}{32}$ in. diameter holes, ¼ in. on centers.

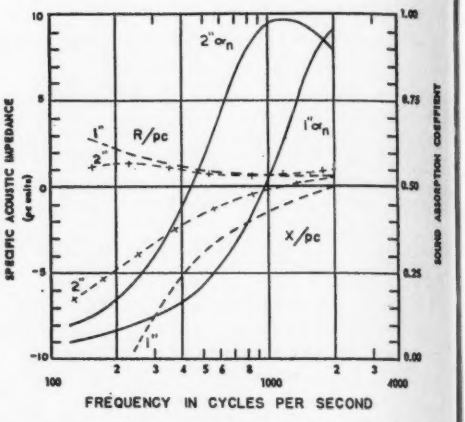

FIGURE 2. Effects of thickness of synthetic fiber batt on acoustic impedance and normal absorption coefficients. Samples were 1 in. and 2 in. thick batts of Orlon, 4.9 lb per ft³, faced with perforated plastic plate same as in Figure 1.

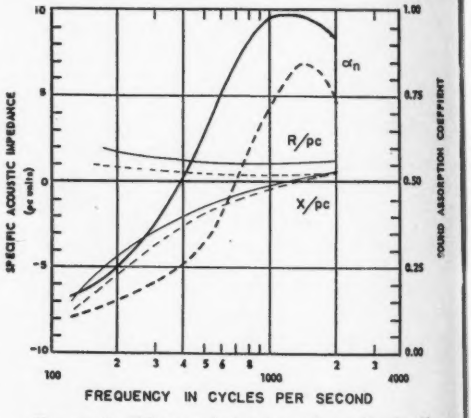

FIGURE 3. Effects of density of synthetic fiber batt on acoustic impedance and normal absorption coefficients. Samples were Orlon packed to 3.5 lb per ft³ (solid lines), and 1.5 lb per ft³ (broken lines), both samples faced with perforated plate same as in Figure 1.

variables are held constant. Figure 3 illustrates the effects of changing the densities to which the fibers are packed. An increase in density increases the resistive impedance somewhat, but has only a small effect on the reactive impedance. Density changes effect sound absorption significantly at the lower frequencies as can be seen by comparing Figures 1 and 3.

Although the absorption coefficients shown in these graphs do not appear particularly high in the lower frequency ranges, it should be noted that the coefficients are measured for normally incident sound waves. Reverberation chamber measurements customarily reported by acoustical materials manufacturers apply to sound randomly incident from all directions and generally exceed the normal coefficients by significant amounts near the cutoff frequency. Normal absorption coefficients for a number of materials have been compared to the reverberation coefficients for the same materials in order to derive empirical conversion factors. In the case of relatively thin (½ in. to 2 in. thick) fibrous blankets of low flow resistance, such as the synthetic fiber blankets just illustrated, it appears that the reverberation chamber coefficients may be approximated by reducing the frequency scale under the normal absorption coefficient plot by one-half, or by considering the normal coefficients equivalent to the reverberation coefficients for a sample half as thick. This relationship between normal and reverberation coefficients has also been described by Koyasu.[4]

Urethane Foams: The importance of flow resistance in controlling sound absorption by urethane foams is illustrated in Figure 4. Increased flow resistance produces increased resistive impedance. Moderately high flow resistance foams will therefore have better low and middle frequency absorption than will low flow resistance foams. However, for thicker samples the optimum flow resistance decreases. Closed cell foams with high acoustic flow resistance have poor sound absorption properties.

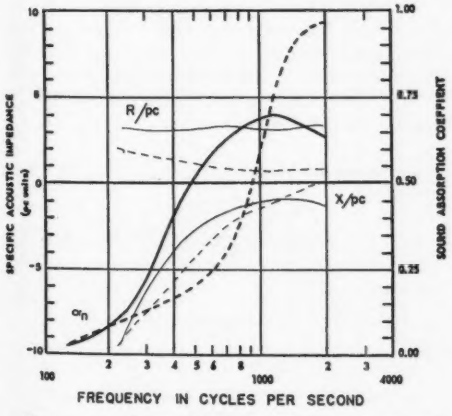

FIGURE 4. Effects of flow resistance on normal absorption coefficients and specific acoustic impedance of typical resilient urethane foams. Samples were 1 in. thick, flow resistances were 675 rayls (solid lines) and 34 rayls (broken lines).

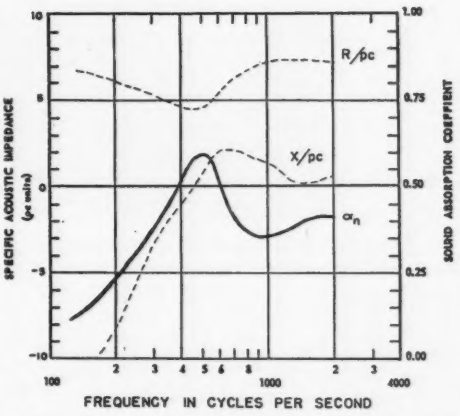

FIGURE 5. Normal absorption coefficeints for a flexible polyvinyl chloride foam with high flow resistance (2000 rayls). Coupling of sound waves to foam skeleton causes absorption peak at structural resonance near 500 cps. Sample thickness was 1 in.

Expanded Polyvinyl Chloride Foams: Certain flexible polyvinyl chloride (PVC) foams exhibit pronounced structural resonances at frequencies within the region of interest. Moderately high flow resistance foams may therefore have unusual absorption curves as shown in Figure 5. The resonance peak similar to that illustrated in Figure 5 at 500 cps can be adjusted somewhat by altering flexibility or thickness of the sample, or it may be made to disappear entirely by reducing flow resistance as shown in Figure 6.

Applications

PROCESS MIXER

An enclosure to control noise was to be constructed around a noisy process mixer running at high speed. Desired noise reduction could only be obtained if a sound trap with a high attenuation could be installed around the feed inlet. Prevention of contamination of the high purity product was a prime requisite in this application. In addition, to be acceptable, the control measures could not interfere with operation, especially during string-up of the continuous strip feed. Without major revisions the inlet sound trap would be limited to about 2 feet in length. A steel perforated facing could be used to protect the absorptive material from wear

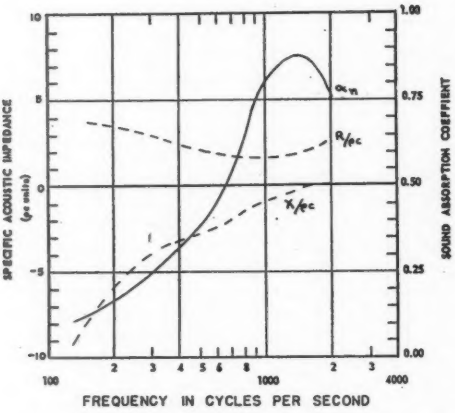

FIGURE 6. Normal absorption coefficients for a flexible polyvinyl chloride foam, 1 in. thick, with moderate flow resistance (250 rayls). Comparison with Figure 5 shows the effect of flow resistance in determining shape of the sound absorption curve.

due to direct contact with the feed. Strong vibrations and static charge build-up on the feed made it inadvisable to use conventional acoustical materials which might flake, sift, or be pulled loose to contaminate the product. The requirement of high absorption coefficients from about 300 cps up to about 5,000 cps further limited the choice of absorptive materials. Preliminary tests were performed using a four-inch diameter impedance tube to determine acoustical characteristics of candidate materials.

The use of nylon fibers, which are compatible with the product quality, as an absorptive lining would minimize the product contamination problem. Consequently our investigation centered on developing a suitable lining composed of these fibers. The curve in Figure 7 shows the normal absorption coefficients for a three-inch thick batt of three denier per filament nylon, packed to a density of 1.75 lb/ft^3 and covered by the protective perforated metal. The essential features of the sound trap are illustrated in Figure 8. The 3-inch thick lining represents a practical maximum that could be used without creating a cumbersome-sized sound trap. The mixer inlet before and after installation of the enclosure and feed sound trap is shown in Figure 9. The lower half of the trap is stationary and the upper half slides back to facilitate stringing up the feed. Close-up of the trap in the open position appears in Figure 10.

Selection of special absorptive material for this problem was based primarily on product contamination considerations. Use of nylon fibers offered a satisfactory acoustical and contamination-free solution.

BLOWER DISCHARGE MUFFLER

A muffler providing 20 db attenuation at 360 cps was needed at the discharge of a blower handling 40,000 cfm of a moisture saturated corrosive gas stream at 80°C. System requirements limited allowable pressure drop through the muffler to 2 inches of water and restricted the muffler length to 8-½ feet. The corrosive nature of the gas stream further limited the choice of materials. A muffler especially developed for

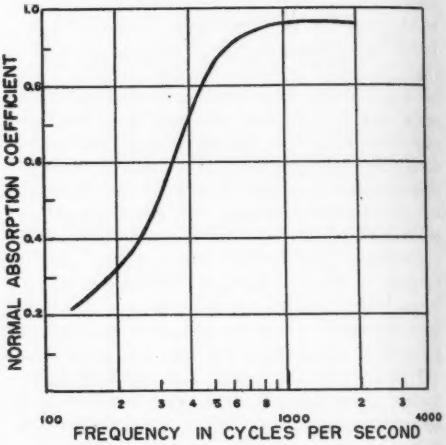

FIGURE 7. Normal absorption coefficients for the 3 in. thick batt of nylon used to line the feed inlet sound trap on a noisy process mixer. The nylon is packed to 1.75 lb per ft^3 and protected by a ⅛ in. thick stainless steel perforated facing with $^3/_{16}$ in. holes, $^5/_{16}$ in. on centers.

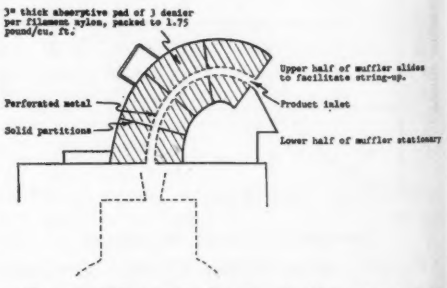
3" thick absorptive pad of 3 denier per filament nylon, packed to 1.75 pound/cu. ft.
Upper half of muffler slides to facilitate string-up.
Perforated metal
Product inlet
Solid partitions
Lower half of muffler stationary

FIGURE 8. Internal details of inlet sound trap for noisy process mixer. The continuous strip feed enters the trap at the right and passes into the enclosure around the mixer at the bottom of the sketch.

FIGURE 9. Inlet before and after enclosure. The continuous strip feed can be seen entering the unenclosed inlet in the left hand picture. The enclosure and sound trap are shown in place in the right hand picture.

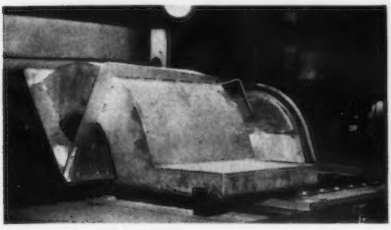

FIGURE 10. Close-up view of sound trap from back with upper half moved back to permit string-up of feed and showing slide runners. Extruded rubber seal can be seen at the far edge of the trap where the upper and lower parts meet. Note perforated facing in lower half (at right). Mixer inlet, at lower right does not show.

this application, and which satisfactorily met the above requirements, has been described previously by Hill.[5] The selection of Orlon fibers for the muffler pack provides an interesting example of the use of special sound absorptive materials.

To provide the necessary attenuation, within the system limitations, a dissipative muffler with an absorptive pack possessing a 95 per cent absorption coefficient at 360 cps was needed. In addition, this pack would have to perform equally well when either soaking wet or dry. Orlon fibers were selected due to their chemical resistance, ability to withstand the abuse of the high air velocities, and high sound absorption coefficients when wet or dry. Sound absorption by a six-inch thick layer of Orlon, both wet and

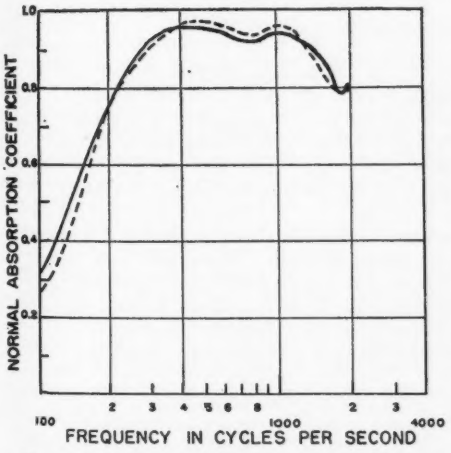

FIGURE 11. Normal absorption coefficients for a 6 in. thick batt of 15 dpf Orlon fibers packed to 2.2 lb per ft³ and faced with a 3/32 in. thick perforated plastic plate with 3/32 in. holes, ¼ in. on centers. The solid curve is with the Orlon dry and the dashed curve is the absorption when the batt was soaking wet. Absorption is shown to be unaffected by addition of moisture.

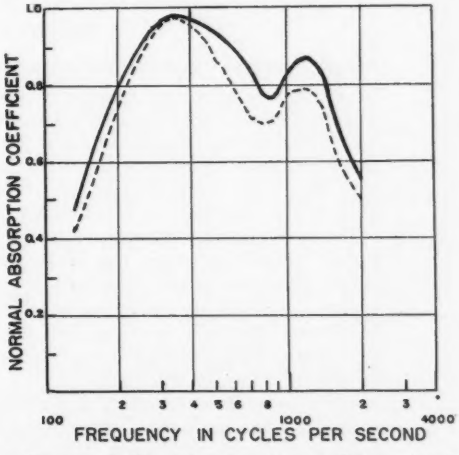

FIGURE 12. Normal absorption coefficients for 6 in. thick batt of Orlon with a resonant facing. Fibers were packed to 1.65 lb per ft³, faced with ¼ in. thick needled batt (felt-like material) and 3/32 in. thick plastic perforated plate with 5/32 in. holes on ½ in. centers. The solid curve is with the Orlon dry and the dashed curve is the absorption when the batts were soaking wet.

dry, is shown in Figure 11. Further increase in sound absorption at 360 cps was obtained by facing the low density Orlon pack with a ¼-inch thick felt-like material before covering with a perforated plastic sheeet. Absorption by such an arrangement is shown in Figure 12. Note that addition of moisture slightly reduced sound absorption by this modified pack, probably due to retention of the moisture in the high density felt. Since the addition of felt would be an added precaution against erosion of the pack by the high air velocities, this arrangement was adopted for the muffler lining.

As a result of favorable experience with the first muffler, two additional mufflers were constructed for similar blowers. These mufflers have operated satisfactorily for over a year with no significant corrosion, erosion, or reduction in the original attenuation.

Summary

In our work it has been found desirable to utilize as sound absorptive media certain materials not generally considered for acoustical applications. Choice of materials which were used depended on certain physical requirements imposed on the sound absorptive material due to a corrosive environment, mechanical abuse, presence of moisture, or product contamination considerations. Use of a commercially available 4-inch impedance tube has proven satisfactory for evaluating acoustical properties of the candidate materials.

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Special Fellowships

THE ATOMIC ENERGY COMMISSION will award a number of fellowships leading to the master's degree in industrial hygiene for the school year 1960–61. Applicants must have a bachelor's degree with major in physics, chemistry or engineering. Mathematics through calculus is required and college biology is desirable. Additional academic training or industrial hygiene experience is also desirable. Fellows must be United States citizens, under 35 years of age and acceptable for graduate work at the University to which assigned.

The tenure of the fellowship is for the academic year. The basic stipend is $2500 with additional allowances for spouse and dependent children. Tuition, fees, and required travel are paid by the fellowship. Fellows having acceptable graduate work or experience may be eligible for an additional $200 in the basic stipend. Fellows will be assisted financially to attend the annual meeting of the American Industrial Hygiene Association. Fellows are expected to devote full time to study and research during the tenure of the fellowship.

Fellows may receive training in industrial hygiene at Harvard University, the University of Cincinnati, the University of Michigan, or the University of Pittsburgh. Assignment to the institution of the fellow's choice will be made when possible but cannot be guaranteed.

Fully detailed information and application materials are available from Industrial Hygiene Fellowship Office, Oak Ridge Institute of Nuclear Studies, Oak Ridge, Tennessee. Applications must reach the Institute not later than March 1, 1960. Appointments will be made on or about April 1, 1960. If you are interested, act at once.

Inhalation Toxicity of Cobalt Hydrocarbonyl*

EDWARD D. PALMES, NORTON NELSON, SIDNEY LASKIN and MARVIN KUSCHNER

Institute of Industrial Medicine, New York University-Bellevue Medical Center, New York City

ALTHOUGH COBALT has had many industrial applications, its use as a catalyst in the oxo synthesis is relatively recent. During this process cobalt carbonyls, the actual catalysts, are formed by the reaction of synthesis gas (carbon monoxide and hydrogen) with cobalt. The industrial operations are carried out in closed systems; nevertheless the absence of information on the toxicity of these materials as well as the possibility of exposure through accident or during maintenance suggested the desirability of a study of their physiological effects. As will be shown, the carbonyl of prime interest, cobalt hydrocarbonyl, decomposes so rapidly in air that in most instances practical exposure is to inorganic cobalt compounds; accordingly these results may well be significant wherever cobalt dusts occur.

There are three cobalt carbonyls: $HCo(CO)_4$, cobalt hydrocarbonyl, a gas at room temperature, $[Co(CO)_4]_2$, dicobalt octacarbonyl, a solid with a vapor pressure of about 0.1 mm Hg at room temperature, and $[Co(CO)_3]_4$, tetracobalt dodecacarbonyl, a solid of very low vapor pressure. In these studies cobalt hydrocarbonyl was selected, because of its high volatility, as the test cobalt carbonyl and was used exclusively in all the work herein reported. The exposures were all by inhalation. The chemistry of the hydrocarbonyl was investigated to the extent required for producing and measuring the test environments, and as background for understanding the biological handling of inhaled cobalt.

The investigation included both single acute inhalation exposures and repeated daily exposures to cobalt hydrocarbonyl. The only previous inhalation work on cobalt carbonyls was that on dicobalt octacarbonyl reported by Armit[1] in 1909. The results of that study will be discussed later.

Kinkaid, Strong and Sunderman[2] examined the toxicity of orally, percutaneously and subcutaneously administered dicobalt octacarbonyl. Their results indicate a low toxicity for the material by these routes. This is not considered to be relevant to the inhalation toxicity of either this material or of cobalt hydrocarbonyl.

* Contributions to the support of this research were made by Esso Research and Engineering Company, Gulf Research & Development Company, Standard Oil Company (Indiana), and Union Carbide Chemicals Company.

Materials

Cobalt hydrocarbonyl was made by acidification of pyridinium cobalt carbonyl $C_5H_5NHCo(CO)_4$ with sulfuric acid, the reaction proceeding as follows:

$$C_5H_5NHCo(CO)_4 + H^+ \rightarrow HCo(CO)_4 + C_5H_5NH^+ .$$

Pyridinium cobalt carbonyl, the pyridine salt of cobalt hydrocarbonyl, was received as a 0.75 to 1.0 molar solution in pyridine; it was stored in screw cap bottles under carbon monoxide. The solution was clear and reddish brown in color. The pyridinium salt in pyridine was kept for months in the refrigerator in the absence of air without apparent deterioration. Bottles opened in air showed decomposition with the formation of a heavy black precipitate.

Experimental Methods and Procedures

For chemical characterization of cobalt hydrocarbonyl a batch method was employed. Sulfuric acid (9*N*) was deaerated by bubbling N_2 through it. A leveling bulb was filled with the acid, capped with a serum stopper and connected by rubber tubing to a reservoir bottle which served for pressure relief. The pyridinium cobalt carbonyl was then injected, by means of syringe and needle, through the serum stopper. On addition of the salt to the acid there is bubbling of the liberated gas and formation of a black residue. Gas samples are withdrawn by another syringe and needle which have been flushed with N_2. The production of $HCo(CO)_4$ is not efficient or consistent by this batch method. Its use was limited to obtaining material for chemical characterization of the decomposition of $HCo(CO)_4$ in air. A much more efficient production method is reported by Sternberg, et al.[3], in which the $HCo(CO)_4$ is swept out of the acid by CO and collected in a cold trap.

The continuous generation procedures is based

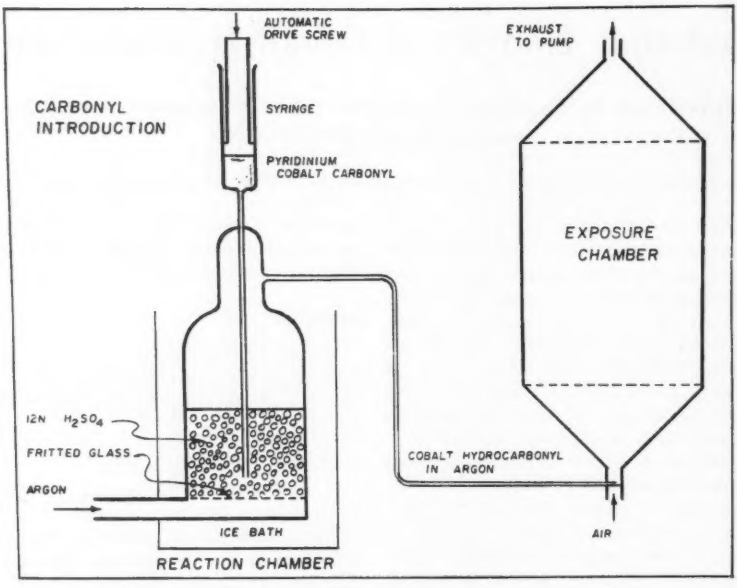

FIGURE 1. Cobalt hydrocarbonyl inhalation exposure system.

on the acidification of pyridinium cobalt carbonyl and scrubbing out of the hydrocarbonyl with an inert gas. The apparatus is shown schematically (not to scale) in Figure 1. At the top there is a motor driven syringe for dispensing at a measured uniform rate a solution of the cobalt hydrocarbonyl in pyridine. The drive screw is powered by a synchronous motor. The pyridinium cobalt carbonyl passes from the syringe through a polyethylene tube into a gas washing bottle with a fritted glass gas disperser. The gas washing bottle of 500 ml capacity is loaded with 200 ml of 9–12*N* H_2SO_4 and is immersed in an ice bath. The pyridinium salt is converted to free gaseous cobalt hydrocarbonyl by the sulfuric acid. Argon is bubbled through the system at a fixed rate and sweeps the hydrocarbonyl out of the acid. Cobalt hydrocarbonyl gas in argon then passes through another polyethylene tube to the exposure chamber just at the air inlet in order to minimize decomposition.

Several exposure chambers were used for the acute studies; these ranged in size from 10 to 20 liters. A critical factor in the acute runs was the rate of chamber ventilation relative to its volume. The reason for this was that the extent of the chemical breakdown of the hydrocarbonyl in air is a function of time. The flow through the chambers for all the acute runs was kept at a value which would give an air flow of one chamber volume in one minute.

The repeated exposures are all conducted in a chamber whose volume was about 1.5 cubic meters. The flow through the chamber was maintained at 300 liters per minute (lpm) to give an air flow of one chamber volume in five minutes.

Chamber air was sampled for particulate cobalt compounds routinely on both the acute and chronic studies. Simultaneous determination of gaseous cobalt compounds was carried out routinely in the acute studies but only occasionally in the chronic exposures, because the concentration of gaseous cobalt in the chamber with the slow air turnover (5 min/change) was negligible whereas the concentration in the more rapidly ventilated (1 min/change) chamber used for acute runs showed low but measureable levels of gaseous cobalt compounds.

The following is an adaptation of the method described by Wise and Brandt.[4] The method was applied to analysis of atmospheres of chambers into which cobalt hydrocarbonyl gas has been introduced. In these studies, cobalt is the only metal present; consequently, there is no problem of interference. The authors in the above report discuss interfering metals and the elimination of some of them. The airborne materials in the present work are gases and finely divided

solids; solution of the sample for analysis offers no problem. The reagents are as follows:

Cobalt standard, 0.001*M* $CoCl_2 \cdot 6H_2O$

Dissolve 238 mg $CoCl_2 \cdot 6H_2O$ in 1000 ml water. This contains one micromole (.0589 mg) Co^{++}/ml; appropriate dilutions are made from it.

Sulfuric acid, 0.02*N*

Phosphate buffer, pH 7.4

Make up a solution of 0.5*M* KH_2PO_4 and 0.4*M* NaOH by dissolving 68.0 g KH_2PO_4 and 16.0 g NaOH in water and bring to one liter.

2-Nitroso, 1-naphthol, 4-sulfonic acid reagent

Make up a 0.001*M* solution by dissolving 253 mg in 1000 ml water.

Apparently keeps well for months without special precautions. (This chemical is obtainable from G. Frederick Smith Co., P.O. Box 1611, Columbus, Ohio.)

The procedure for the analysis is as follows: To sample or aliquot in 4 ml of 0.02*N* H_2SO_4* or water or mixture of acid and water add one ml of the nitroso reagent plus 5 ml buffer. Color develops almost immediately and is stable for hours. Read in spectrophotometer at 525 millimicrons.

Calibration curves using a Coleman Jr. Spectrophotometer with the macro cuvettes (about 1.6 cm path length) were run frequently. A composite calibration curve is shown in Figure 2. All values are corrected for the blank run in the same series; the optical density values for the blank averaged 0.063 O. D. units with a standard deviation of 0.005 when read against water. It is seen that Beer's law is obeyed up to about 10 μg Co^{++} but the curve flattens beyond this point. About 15 μg apparently exhausts the dye so that no further color is produced by addition of more Co^{++}. With larger quantities of dye larger amounts of cobalt can be determined, but the blank value increases correspondingly and no improvement is seen in the useful range of the test.

This procedure has been used for measuring concentrations of particulate and gaseous cobalt compounds in the exposure chambers. It should be made clear that there has been no necessity to examine interfering materials and the test should not be applied to field use without such investigation.

Particulate cobalt is collected on ½-inch discs of Whatman No. 41 filter paper held in a Swinny Filter holder (Aloe Cat. No. 42360). This is a very compact unit intended for use in Seitz filtration of parenterally injected fluids. Collection efficiency increased with the dusts sampled up to about 700 ml/min. The effective sample collecting area is about 8 mm in diameter. Thus the linear velocity at 700 ml/min. was 23 cm/sec; it is suggested that at least as high a linear velocity be maintained if another diameter paper is used. Efficiency, based on two papers in series, was above 95 per cent. (Other sampling methods tested during the repeated exposures showed millipore filters, Cambridge Absolute Filters and a modified electrostatic precipitator to be as good or better than the Whatman No. 41 for sampling. The midget impinger on two occasions showed collection efficiencies less than 10 per cent that of the Whatman No. 41. Theory, of course, requires that particulates of this size be difficult to impact.)

The filter paper is transferred to a 15 ml centrifuge tube containing 4 ml of 0.02*N* H_2SO_4. The solution is mixed by tilting the tube. The dusts go into solution readily and the filter paper has been left almost completely intact; thus centrifugation is not usually necessary. If, however, solution is not accomplished readily and the filter paper is macerated to aid solution, the suspension is spun in a centrifuge. Up to the entire 4 ml can be used or an aliquot can be taken. It is possible to develop the color in the presence of the filter paper.

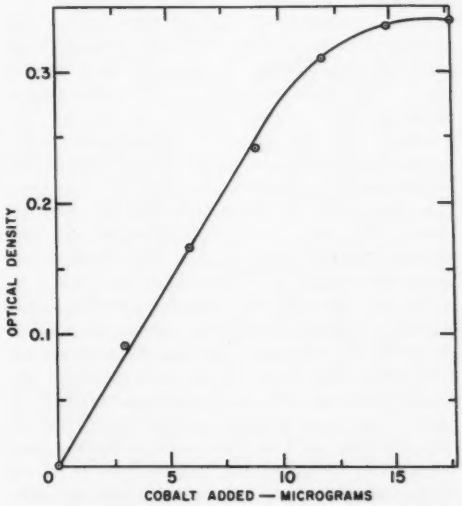

FIGURE 2. Cobalt calibration curve using 2-nitroso, 1-naphthol, 4-sulfonic acid reagent. All values corrected for blank.

* This quantity of H_2SO_4 has been used to dissolve filter paper samples routinely. The buffer can take care of up to about one milliequivalent of acid without significant interference in the color development.

Gaseous cobalt is collected by a bubbler in the sampling line following the filter paper unit. Sintered glass gas dispersion tubes (Corning Cat. No. 39533C) are used in a 25 × 200 test tube fitted with a two-holed rubber stopper. The sampling liquid is made up of 4 ml water + 1 ml 0.001*M* 2-nitroso-1-naphthol-4-sulfonic acid + 5 ml of buffer. At the end of the sampling period the bubbler is disconnected, a syringe is connected to the top of the dispersion tube by rubber tubing and the solution is mixed by pumping back and forth so that the top of the liquid rises an inch or two above the frit each time. This solution is then poured into a cuvette and read.

Rats in the excretion studies were kept in metabolism cages except when they were actually being exposed. Thus urine was collected for 16-17 hours on exposure days and 24 hours on other days. The cages were designed to minimize food spillage and spilled water was diverted outside the cage. The urine was collected in flasks and its volume measured. The large glass funnel of the metabolism unit was then washed into the container and the combined urine plus washings were ashed and analyzed together. Feces were caught in a screen covering the funnel and were discarded. The results were calculated as mg Co/rat/day; the reason for this mode of expression was the difficulty of estimating precisely the urine volume considering the possibilities for holdup. The washings from the funnel contributed, on the average, about 25 per cent of the cobalt found. This fraction was consistent with losses that might be incurred with holdup and drying of urine on the funnel. In any event the washings represented a minor portion of the total cobalt and could not lead to gross errors in interpretation.

Dogs were also kept in metabolism cages. Urine, feces, food and debris all fell through the expanded metal floor of the cage into the stainless steel pan. The urine collected in a depression in the pan. A stopper in the spout on this portion was removed and the urine drained off. Feces were discarded. Since there was some concern that the shed hair which collected in the urine pan might contribute a significant amount of cobalt to the urine, the following procedure was used. That part of the pan in which the urine stood (a very small fraction of the total area) was washed with a definite volume of dilute HNO_3 and this was drained into the container with the urine. The total volume was then measured and urine volume obtained by subtracting the volume of wash acid used. The whole pan was then washed thoroughly with dilute HNO_3 so that all cobalt would be extracted from any debris in the pan; this washing was analyzed separately. It was found to contain just about as much cobalt as did the urine collected over the same period. Several attempts were made to eliminate this difficulty, including a shampoo for all the dogs immediately following the last exposure; on this occasion the dogs were lathered and rinsed twice. The cobalt in the cage debris dropped only to about half its pre-treatment level. One experiment conducted while the dogs were on test gave a clear-cut answer on the question of contamination of the urine by hair, etc. One day three of the four dogs on being returned to the cages all voided within a few minutes. These uncontaminated specimens were analyzed separately from the rest of the day's collection made in the normal way. Analytical values for the two specimens showed 3.7 mg/l for the fresh and 3.5 mg/l for the remainder of the day's urine. In short it can be stated that although the potential for contamination was high, the actual contamination appeared to be negligible.

Lungs of rats were removed as described elsewhere and placed in 3 per cent formaldehyde. After fixing, sections were taken for histologic examination and the remainder of the lungs plus the formaldehyde solution was ashed and analyzed in the same manner as the urine.

The analytical procedure adopted here was one used by Dr. Carl A. Nau[5] and Mr. Jack Neal; it was based on a communication from Mr. Dohrman H. Byers. The method suggested by Dr. Nau is as follows:

Determination of small amounts of cobalt in urine:

"Transfer by means of a pipette a 2 to 100 ml sample of the well mixed specimen of urine to a 50 ml beaker for 25 ml samples or less, or a 250 ml beaker for larger samples. (The sample is so chosen to give less than 40 gamma of cobalt. Gamma = 0.001 milligram.) Evaporate just to dryness of a hot plate, cool, add from 2 to 20 ml of HNO_3 (sp. gr. 1.42), depending on the amount of sample taken, and again evaporate to dryness. Transfer the beaker containing the dried residue to a muffle furnace maintained at 500°C and ignite in air enriched with oxygen until the residue in the beaker is white. This usually requires 5–10 minutes. Cool, add 2 to 20 ml of HCl (sp. gr. 1.18), and again evaporate just to dryness. Cool and add 19 ml of acid solution (25 ml of HCl (sp. gr. 1.19) and 5 ml of HNO_3 (sp. gr. 1.42) diluted with sufficient water to make one liter). Evaporate to 7 ml. Add 2 ml of a 0.1 aqueous solution of Nitroso-R salt and 2 g ±

0.1 g of sodium acetate. Heat to boiling and boil for 45 seconds. Add 1.5 ml of HNO_3 (sp. gr. 1.42) and boil for 45 seconds more. Cool to room temperature in the dark and dilute to 35 ml with cold water. Compare the red color produced with that of standards taken through all steps of the method. A blank including all the reagents should be carried through and any cobalt found deducted."

At this laboratory the final colors were read on the Coleman Jr. Spectrophotometer at 510 millimicra. Calibration curves for pooled urines of unexposed rats with cobalt added are shown in Figure 3. It is seen that 1 to 7 mg/l (10 to 70 μg/10 ml) of Co can be easily determined in a final volume of 35 ml. The blank values for rat and dog urine were virtually identical.

Whole rat lungs were analyzed by the same procedure.

Only rats were used on the acute studies. Male albinos of 100 to 250 g were used. They were obtained from Blue Spruce Farms and held for a minimum of one week before use. They were fed Purina Lab Chow and water ad libitum. The rats were housed three to five to the cage. Those showing evidence of poor condition such as weight loss, noisy breathing or diarrhea were not used.

Two preliminary runs were made to aid in the selection of a cobalt concentration level for the three months exposure. The animals in these runs were treated as in the acute studies. The following is the description of animal care and procedures for the three-month study.

Rats, male albinos, were received as recent weanlings (ca 90 g) and kept for six weeks before the experiment began. They were housed five to the cage for the first four months and thereafter three to the cage. Having been numbered on arrival, they were randomized by card shuffling and redistributed to their permanent cages. Any animals in poor condition were discarded at this point. Thereafter rats were weighed once per week through the second week after the end of exposure and monthly thereafter. All animals dying or sacrificed on or after the test were autopsied. Hematology was done on all animals before randomization, near the end of exposure, and just before sacrifice for those serially sacrificed. Air conditioning sufficed to maintain the temperature of the exposure chamber below 85°F. Animals were not fed or watered during the exposures.

Male guinea pigs were received at 350–450 g twelve weeks before the test started. They were housed four to the cage for living and for exposure. They were fed Rockland Guinea Pig Diet and water ad libitum without any supplement. All other observations and procedures were the same as for the rat.

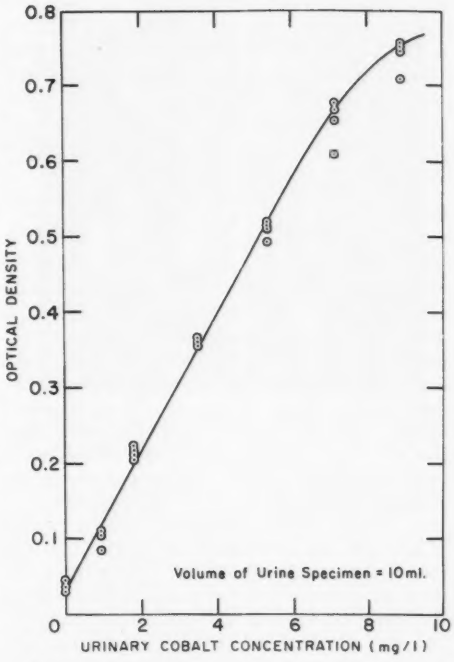

FIGURE 3. Cobalt added to rat urine-nitroso R-salt reagent.

Dogs, two male and two female thoroughbred beagles, were obtained from the Roscoe B. Jackson Memorial Laboratories at Bar Harbor, Maine. These animals were litter mates, whelped seven months and received three months before the exposure started. The diet was Allied Mills Kibbled Krumbs and Arrow Boiled Horsemeat. The dogs were fed at the end of each day's exposure. They were housed in individual cages for living and both animals of the same sex were exposed in the same cage. Otherwise, the dogs were on the same regimen as the other animals.

One of the important aims of the acute work was to establish whether or not there was a significant risk of pulmonary edema with $HCo(CO)_4$. Lung to body weight ratio was chosen as the most satisfactory and objective criterion on which to base an estimate of pulmonary edema. It should be added that the edema would be expected to manifest itself maximally about 18–24 hours after exposure. For this reason, most of the acute animals were sacrificed 24 hours

after exposure. All species were sacrificed by intraperitoneal Nembutal. When the animal was in deep anesthesia the chest wall was opened and the heart and lungs dissected out while the animal was still alive. The heart was removed, the trachea cut off just above the bifurcation, any blood gently blotted off, and the lung weighed to 5 mg on a previously tared paper.

Results

Chemistry

Relatively concentrated mixtures of $HCo(CO)_4$ gas were prepared by the batch procedure described. The gas liberated by acid was introduced into an evacuated 10 cm infrared gas cell and its decomposition measured by following the decrease in optical density at the appropriate bands in the 5.0 micron region. The gas is relatively stable in the absence of any diluting gas or in an atmosphere of CO. Figure 4 shows its decomposition in air or N_2. These experiments were carried out by introducing a known volume of gas into the evacuated cell and setting the wave length of the spectrophotometer at one of the absorption maxima for cobalt hydrocarbonyl. At zero time the cell was opened to air or N_2 and then closed. Optical density was then followed as a function of time. In the two examples shown it is estimated that there was about 1.5 per cent gaseous hydrocarbonyl at zero time. It is seen that about 80 per cent of the hydrocarbonyl remained after 2.5 minutes' exposure to N_2, while the decomposition in the presence of air was virtually complete at less than two minutes.

Thus, the hydrocarbonyl is quite short lived in the presence of oxygen; the course of degradation and the end products have not been defined.

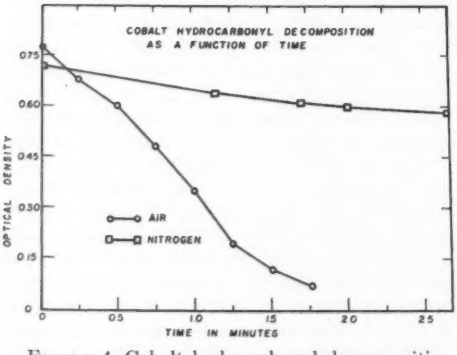

FIGURE 4. Cobalt hydrocarbonyl decomposition as a function of time.

It is possible to make a crude estimate of the amount of carbon monoxide released in the decomposition from the infrared spectrum. Such an estimate gives a ratio of about five moles of CO for one mole of the hydrocarbonyl; this is a value not far from the theoretical yield of four moles per mole for the complete degradation of $HCo(CO)_4$. Since there is considerable uncertainty as to the molar extinction coefficient for the hydrocarbonyl and furthermore since the intensity of carbon monoxide absorption is very low at the concentrations involved, these estimates are approximate.

Additional evidence has been secured by conductivity measurements confirming the view that cobalt hydrocarbonyl exposed to air decomposes within a few minutes. Gas sampled from the argon line of the cobalt hydrocarbonyl generator was found when dissolved in water in the absence of air to display electrical conductivity of the degree anticipated, namely about the same as that of an equimolar solution of sodium chloride. Such conductivity is to be expected since the hydrocarbonyl is a strong acid.[3] After the hydrocarbonyl was mixed with air, no conducting ions could be detected.

The proportion of cobalt in gaseous forms was checked both in the argon stream and in the chamber by sampling with the filter-bubbler combination, as described above. Samples collected from the argon line showed practically no particulate cobalt; however, after being in the exposure chamber for a matter of a few minutes the situation was completely reversed in that particulates now made up at least 90 per cent of all the cobalt.

The chemical composition of the particulates from the exposure chamber has not been definitely established. Analysis in two instances gave crude estimates of the cobalt content of the airborne particulates, yielding in both cases approximately 50 per cent cobalt. Compounds of cobalt which are considered possible along with the percentage of Co in each are:

CoO	78.7%	$Co_3O_4 \cdot 3H_2O$	60%
Co_3O_4	73.4%	$Co_2O_3 3H_2O$	53.6%
Co_2O_3	71.1%	$CoCO_3$	49.6%

Thus it appears that the cobalt contents of the carbonate or one of the hydrated oxides are consistent with the rough analytical values obtained. The evidence is not considered sufficiently strong, however, to draw any definite conclusions as to the identity of the anion or anions.

X-ray diffraction studies performed by Mr. N. Talvitie of the Occupational Health Program,

USPHS, Cincinnati, indicated a lack of detectable crystal structure.

The presence of either dicobalt octacarbonyl or tetracobalt dodecacarbonyl in the chamber was an extremely poor possibility simply from mass action considerations. This stems from the large dilution in the chamber. In order that the hydrocarbonyl be converted to the dimer $[Co(CO)_4]_2$ it would be necessary that the pairs of molecules of hydrocarbonyl come together, probably an infrequent occurrence considering the distances between the molecules after dilution. The possibility of forming the tetramer $[Co(CO)_3]_4$ was considered so remote as to be negligible.

It is concluded, therefore, that the animals were exposed primarily to simple inorganic cobalt hydrocarbonyl decomposition products.

Particle size was measured on an electron microscope at 10,000–14,000 power. Samples were collected on graphite grids which were placed in the collecting cylinder of an electrostatic precipitator. The grids were shadowed and photographed. The plates were placed in a photographic enlarger and further magnified by five times and individual particles were measured with a ruler. A reproduction of one of the electron photomicrographs is shown in Figure 5. It is seen that the majority of the particles appear to be aggregates; the overall median size is 0.1 micron diameter or 0.3 micron mass median diameter. There are many particles of about 0.01 micron diameter.

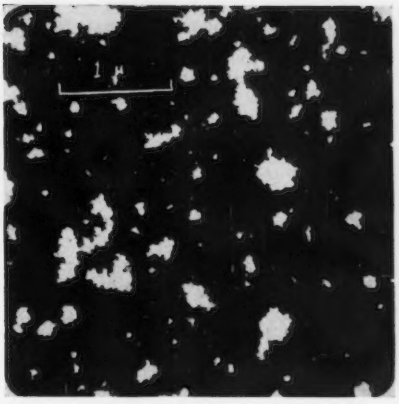

FIGURE 5. Electron photomicrograph of solid decomposition products.

Toxicology

The results of the inhalation studies are covered in three sections, (a) the two week mortality, etc., following single exposures to cobalt hydrocarbonyl and its decomposition products, (b) assessment of acute pulmonary irritation produced within 24 hours after single exposures to the hydrocarbonyl, and (c) the toxicity of repeated inhalation of the hydrocarbonyl at a low concentration.

The results of observation of rats for two weeks following exposure to cobalt hydrocarbonyl for 30 minutes are shown in Table I. The control animals were exposed under identical conditions as the test groups except that there was no pyridinium cobalt carbonyl in the pyridine metered into the generator in the control runs. To check the possibility that pyridine or sulfuric acid mist from the generator might be present in toxic concentrations, control runs were made in which the pyridine was metered in the same way as the pyridinium cobalt carbonyl in pyridine. Chemical determinations of the generator output were made for pyridine and H_2SO_4; the concentration in chamber air was never as high as the MAC for either of these materials on any experiment.

After exposure the survivors showed a response typical of pulmonary irritants. The affected animals during exposure showed labored or otherwise disturbed respiration as the most pronounced clinical sign. As noted in the table, all of the deaths occurred during the exposure or within three days thereafter; this is entirely consistant with death due to acute pulmonary irritation. The two-week LC_{50} is estimated from these data to be 165 mg Co/M^3.

These results should be compared with those of Armit[1] for dicobalt octacarbonyl (called cobalt tetracarbonyl by Armit). To permit com-

TABLE I

Single 30-Minute Exposure of Rats to Cobalt Hydrocarbonyl

Two week post-exposure observation:

Run No.	Co Conc. (mg/M³)		Mortality dead/total	Survival time, rats dying on test (days)
	Total	Dust		
1A2	0	—	0/5	—
0X9	0	—	1/5	—
1A23	—	7	0/5	—
1A18	—	28	0/5	—
1A3	49	47	0/5	—
1A21	—	68	0/5	—
1A12	—	78	5/5	0,0,0,0,0
1A20	—	113	2/5	1,3
1A4	—	191	4/5	1,1,2,2
1A24	—	245	5/5	0,0,1,3,3
1A5	252	222	1/5	2
1A1	436	408	5/5	0,0,0,0,0

parison it is necessary to present the details of the one experiment which was fully covered in his report. The cobalt carbonyl crystals were placed in a tube maintained at 21.5°C and air passed over them at 0.3 liter per minute. The description adds that,

"The air was then passed through a glass chamber guarded by plugs of cotton wool at the outlet and inlet and thence bubbled through bromine water in a wash bottle and a Meyer's apparatus."

One rabbit was exposed in this run for one hour. Armit found only 0.175 mg Co in the bromine flask and 1.2 mg in the cotton plugs. It can be calculated that 18 liters of air (0.3 lpm for one hour) flowed through the system. If the bromine flask plus the cotton plug on the outlet were considered as a sampler it would be possible to get a maximum value for flask plus outlet plug by simply dividing all the recovered cobalt (1.375 mg) by the total volume of air (18 liters); this gives an upper limit for the concentration of 76 mg Co/M³. In light of the values reported here for the 30-minute rat LC_{50} it is not surprising that the rabbit survived. In their report on toxicity studies of dicobalt octacarbonyl, Kincaid[4] and coworkers in discussing Armit's work use the assumption of saturation at room temperature. The vapor pressure of dicobalt octacarbonyl is 0.07 mm Hg at 15°C.[6] The concentration was, therefore, estimated at 100 ppm which is equivalent to about 470 mg Co/M³. On the contrary, the above calculation shows that the concentration was substantially less than 76 mgm/M³.

Additional light is thrown on this point by some experiments carried out at Carbide and Carbon Chemical Co.[7] on generation of dicobalt octacarbonyl atmospheres for evaluation of sampling methods. It was found that consistent results could not be obtained when air was passed over dicobalt octacarbonyl crystals in a tube. The generating system was apparently identical in principle to that described by Armit above. It was found, however, that uniform results could be secured when CO was substituted for the air.

In our study, groups of animals were sacrificed at 24 hours after exposure and lung/body weight ratios were taken. The results are shown in Table II which gives the cobalt concentration, the 24-hour mortality and the incidence of edema which is here defined as a lung/body weight ratio greater than 0.9 per cent.

The controls showed little edema. A definitely dose-related increase in incidence is seen in the group of animals receiving cobalt. It should be noted that the three groups exposed to 106–137 mg/M³ had an incidence of 11/15.

The last column in Table II shows the incidence of grossly observed lung damage. The signs included as indicative of such damage are edema, congestion, hemorrhage or consolidation of lung areas; presence of one or more of these findings was sufficient to judge the animal positive. It is seen that 5/31 of the control animals were positive indicating an expected incidence of about one positive rat for each group of five without any treatment. The low doses of the hydrocarbonyl did not produce any significant increase but all higher doses did. At all levels greater than 106 mg/M³ all animals exposed to cobalt were positive.

It is worth noting here that lung/body weight ratio provides a simple, objective criterion for evaluation of lung damage from inhaled irritant materials.

Two pilot runs were performed at low concentrations with limited groups of animals to give a basis for planning the repeated exposures. The results are shown in Table III. The first experiment was performed on 10 rats; the Co concentration averaged 26 mg/M³. It is seen that four of the rats died and there were clinical signs of respiratory difficulty and ruffled fur in almost all animals. These results are consistent with those obtained by Carpenter[8] on CoO. He exposed rats, guinea pigs and rabbits for repeated eight-hour periods to 50 mg CoO/M³ (39 mg Co/M³) and concluded that this concentration is much too high to be tolerated without ill

TABLE II

Pulmonary Edema and Gross Lung Damage Caused by Inhalation of Cobalt by Rats

All exposures of 30-minute duration, animals sacrificed 24 hours after exposure.

Run No.	Co Conc. mg/M³	Pulmonary edema*	No. Rats showing gross lung damage**
0X1-0X8	0	2/38	5/31
1A22	7	1/5	1/5
1A17	26	1/5	2/5
1A14	83	0/5	4/5
1A16	90	2/3	2/3
1A15	106	3/5	5/5
1A10	116	2/4	5/5
1A19	137	5/5	5/5
1A11	179	5/5	5/5
1A13	236	1/1	1/1

* A lung/body weight ratio greater than 0.9 per cent is the criterion for a positive diagnosis.

** Any one or more of the following gross autopsy findings are interpreted as gross lung damage: hemorrhage, edema, consolidation, congestion, pleuritis, bronchiectasis, emphysema or atelectasis.

effect. None of the rats survived more than 25 exposures.

Another group of animals was exposed for a total of 53 hours to an average concentration of about 12 mg/M³. It is seen in Table III that there were two deaths but practically no other observable adverse effects during this exposure. The deaths occurred three and eight days after the exposure started. It was concluded that this level would be tolerable for most test animals for three months. Consequently, the three-month exposure was set up with a design level of 9 mg/M³; exposures were six hours per day, five days per week for three months. A total of 71 exposures was performed. The test animals were (at the start of the experiment) 75 rats (males), 35 quinea pigs (males) and 4 dogs (2 male, 2 female). Observations included (a) cobalt concentration of chamber air, (b) mortality, (c) weight and clinical condition of animals, (d) hematology, (e) urinary cobalt excretion by rats and dogs, (f) cobalt concentration in rat lungs, (g) gross and microscopic pathology.

The average chamber concentration for each exposure was calculated from six to twelve individual chemical determinations on each exposure. The average values for individual exposures gave an overall mean concentration of 9.0 mg/M³ with a standard deviation of 1.2 mg/M³. The peak values for each day's exposure showed 10.6 ± 1.2 mg./M³.

The highest single value attained was one of 30 mg Co/M³; this was the result of a brief breakdown in the chamber exhaust.

The particulate portion of the Co in the chamber was so preponderant (more than 99 per cent) that its value was used to express the chamber concentration.

In the repeated exposure experiment, there were totals of 16 deaths out of 75 rats, 3 out of 36 guinea pigs, and none out of 4 dogs. The guinea pig deaths were distributed as follows: one in the week before hydrocarbonyl exposures started (week minus 1), one in the sixth week of exposure (week 6), and the last in the eleventh week of exposure. In the rats there were three deaths in week minus 2, two in week minus 1, eight in week 1 (the first week of hydrocarbonyl exposure), and one each in weeks 4, 8, and 10.

The guinea pig deaths seem randomly distributed in time and probably not dose related. The rats, on the other hand, had an excessive mortality during the first week of hydrocarbonyl exposure. On post-mortem examinations, it was found that all of these rats had lung infections and showed some degree of edema, congestion and bronchitis. The most probable explanation for these deaths in the light of the low mortality observed later is that these were ill animals when put on test and the deaths were the result of inability to withstand the added insult of the cobalt exposure. The mortality in these is considered probably dose related and to be an acute rather than a chronic effect.

TABLE III

Preliminary Repeated Cobalt Exposures

Exp. Series	Avg. Co Conc. (mg/M³)	No. of exposure	Total days elapsed 1st to last exposure	Total hours	Mortality (dead/total)	Survival after first dose (days)	Clinical signs* (No. Pos.)
1C1	26	3	4	9.4	4/10 rats	2,3,7,9	9F 7R
1C2	12	9	11	53.0	2/18 rats	3,8	none
					0/12 guinea pigs	—	none
					0/1 dog	—	R

* Clinical Signs
F—Fur Ruffled
R—Rales or impaired breathing

It was considered desirable to isolate, if possible, the effects of the exposure regimen itself from those produced by the cobalt. To this end the animals were given six sham exposures in the 18 days preceeding the start of actual exposures; five of these six exposures were within the final eight days of the period. All three species adapted to the exposure regimen quite rapidly and were gaining weight when the exposure to hydrocarbonyl was started. It developed that, with one exception, no remarkable weight losses were observed throughout the exposure and six-month post exposure period. The exception was in the dogs in which a consistent loss was observed for the first five weeks of hydrocarbonyl exposure. The dogs then began to gain and continued to do so for the rest of the experiment. Rat and guinea pig weights were compared statistically with those of their unexposed controls during the pre-exposure period, immediately post exposure and three and six months post exposure. The only significant result was a slower weight gain in the rat at the end of exposure ($P < .01$) and at three months post exposure ($P = .05 - .01$). It should be pointed out that at six months post exposure the test rats were not significantly different in weight from the unexposed controls. The differences observed may be attributable to the exposure regimen as well as to the hydrocarbonyl itself. The effect was transitory in that it disappeared following termination of exposure. The weight curves for all three species are shown in Figure 6.

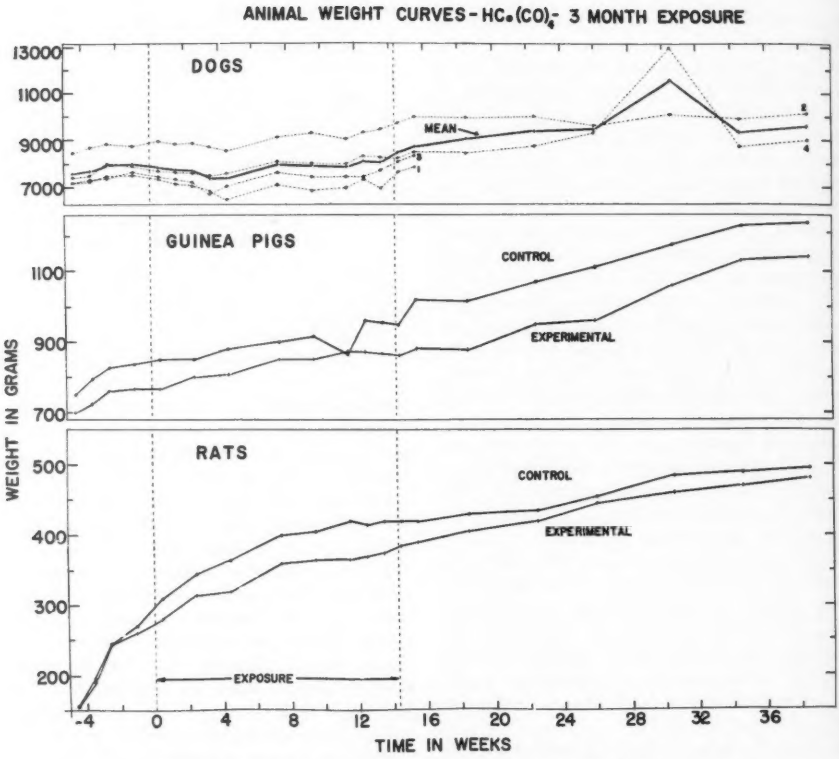

FIGURE 6. Animal weight curves for three-month exposure.

There was nothing unusual in the general appearance of the animals for the duration of the observation period.

The hematological examination of the animals included hemoglobin (Haden Hausser Hemoglobinometer), WBC and differential. All animals, test and control, were examined before exposure, at the end of exposure and six months after exposure. The average values and standard deviations are shown in Table IV. The most striking change is in the rat hemoglobin values. Although it is seen that there is an increase in both control and test groups between the pretest and the end of exposure observations, the increase is much more marked in the test group, the difference being significant at the 1 per cent level. This difference is attributed to the well known hematopoietic effect of cobalt. It is seen that the abnormally high hemoglobin level is still apparent six months after exposure. This is not interpreted as a toxic manifestation but rather a normal physiological response; it might have diagnostic significance, however, as an indication of cobalt exposure. The hemoglobin levels of the guinea pigs were raised significantly but not nearly as markedly as those of the rat.

The hematological findings included also (a) decreased monocytes and increased basophils in the rat test group immediately after termination of exposure, and (b) decreased lymphocytes and increased basophils in the guinea pig test group immediately after exposure. All other hematological findings are considered normal.

Two goals were sought in the measurement of urinary cobalt, (a) the level of excretion established after a month or more of daily exposure to a well characterized cobalt containing atmosphere, and (b) the kinetics of buildup and washout of the inhaled cobalt.

Urines were collected and analyzed for all four dogs during the approximately 18 hours following six of the last nine exposures. The mean values and standard deviations obtained were 4.1 $\pm$ 0.8 mg Co per liter of urine or 0.64 $\pm$ 0.43 mg Co/dog/24 hours. Urines collected from five rats on seven days from the 39th

TABLE IV

Hematology in 3-Month Cobalt Exposure Study

			No. of animals		Hb gms	WBC thousands	Lymphocytes %	Polymorph %	Monocytes %	Basophil %	Eosinophil %
Rats	Before exposure	Control	41	Mean	14.05	10.8	79	17	3.3	0.15	0.8
				S.D.	0.80	2.4	7	6	4.0	0.40	1.2
		Exptl	75	Mean	14.00	11.2	81	16	2.2	0.10	0.5
				S.D.	0.80	3.2	7	6	2.2	0.34	1.1
	Immed. after	Control	34	Mean	15.75[b]	18.4	69	25	5.0[b]	0.06[b]	3.9
				S.D.	0.65	5.6	11	8	2.1	0.23	2.9
		Exptl	57	Mean	17.30[b]	22.4	69	26	2.2[b]	0.32[b]	3.4
				S.D.	1.55	8.0	11	11	2.2	0.50	2.3
	6 Months after	Control	19	Mean	15.70[b]	15.6	56	37	2.4	0.00	4.8
				S.D.	0.50	4.0	9	9	2.7	—	3.4
		Exptl	31	Mean	16.40[b]	17.2	59	33	4.1	0.07	6.5
				S.D.	0.65	5.0	8	8	2.1	0.24	3.4
Guinea pigs	Before exposure	Control	8	Mean	14.49	10.8	75	25	1.6	0.38	0.1
				S.D.	0.51	2.4	13	17	0.8	0.61	0.5
		Exptl	36	Mean	15.00	10.8	65	33	2.0	0.14	1.0
				S.D.	0.80	3.6	26	21	2.0	0.33	2.0
	Immed. after	Control	6	Mean	15.09[a]	10.4	65[a]	30	5.2	0.17[a]	1.6
				S.D.	0.31	4.0	12	12	2.1	0.34	1.4
		Exptl	32	Mean	15.90[a]	14.2	45[a]	40	7.4	1.06[a]	7.2
				S.D.	0.70	5.6	17	22	3.0	0.99	8.8
	6 Months after	Control	3	Mean	14.51	10.0	61	35	3.0	1.33	1.6
				S.D.	1.00	4.4	8	12	1.8	0.98	1.0
		Exptl	15	Mean	15.40	12.4	74	20	2.0	0.20	3.6
				S.D.	0.80	4.6	13	14	2.0	0.27	4.0
Dogs	Before exposure	Exptl	4	Mean	14.5	12.4	48	50	2.0	0.25	0.3
	Immed. after	Exptl	4	Mean	15.0	17.4	38	57	2.5	0.00	3.5
	6 Months after	Exptl	2	Mean	15.2	15.0	36	58	3.5	0.00	2.5

a—Difference significant at 5 per cent level.
b—Difference significant at 1 per cent level.

to the 54th exposures showed a mean excretion of 0.063 ± 0.016 mg Co/rat/24 hours. Considering the rat urine volume, which was not accurately measured, to be about 12 ml per day, this would correspond to about 5 mg Co/liter of urine. These urinary cobalt levels were obtained after repeated exposure to an air concentration of 9.0 mg Co/M³.

The buildup and washout of Co in rats was studied by introducing a previously unexposed group of rats into the exposure chamber for three exposures in one week followed by four more exposures in the next week. Urinary cobalt was determined on the rats before, during and after exposure. The average values are plotted in Figure 7. The convention used in Figure 7 is that the exposure starts the experimental day and urine is collected for the remainder of the day; thus day 0 starts with the first exposure

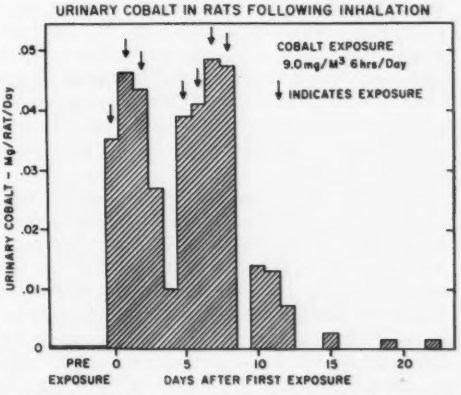

FIGURE 7. Urinary cobalt in rats following inhalation.

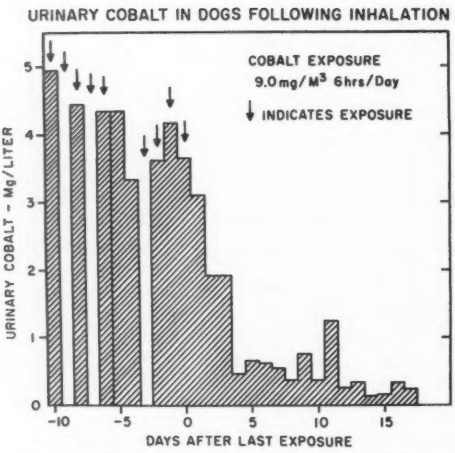

FIGURE 8. Urinary cobalt in dogs following inhalation.

and terminates with the beginning of the second exposure. It is seen that there is a rapid rise in urinary cobalt on the day of the first exposure. A rapid fall is noted for days 3 and 4, a weekend during which the rats were not exposed. Day 5 shows another rapid rise and a modest additional increase is seen during the next three exposures. All samples were lost on day 9, but day 10 closely resembles the previous Sunday (day 3). Thereafter, there is a slow loss of cobalt for at least 14 days after the last exposure. It appears that the Co level drops exponentially at least for a few days after the exposures are stopped. Assuming this to be the case, the time for reduction of the steady state exposure level is 1/2 this value (the half-life of excretable cobalt) is about one day. It is noted that the buildup time is probably even shorter. It appears, therefore, that there is a rapid turnover of the bulk of that portion of cobalt which is excreted in the urine. A portion of the metal may be retained for considerable periods as evidenced by the fact that it is still detectable for two weeks after the last exposure.

A similar study was carried out on the four exposed dogs with the exception that the buildup of urinary cobalt was not followed. The data are presented in Figure 8 as urinary cobalt concentration rather than quantity per day since this gave more consistent values. The dieaway curve is still quite similar to that observed for the rats and also leads to an estimated half-life of about one day. The level of cobalt excretion is still measurable for two weeks following termination of exposure as was observed in the rat. The significance of these trace values was examined by running urines on dogs not receiving cobalt but housed in the same type cages and receiving the same food. The comparison showed that the cobalt excretion of the test dogs was higher than the control dogs ($P = <.01$) at two weeks after cessation of exposure.

An estimate was made of the fraction of inhaled cobalt excreted in the urine. The amount of cobalt breathed in during a six-hour exposure was calculated from the minute volume and the average cobalt concentration for repeated exposures. The first was estimated from the equation[11]: minute volume in liters $= 2.10 \times$ (wt. in grams)$^{3/4}$. This led to estimates of 0.52 mg Co inhaled during a six-hour exposure for the rats and 5.1 mg for dogs. The daily urinary outputs were 0.064 mg/day for the rats and 0.79 mg/day for the dogs. This amounted to 12 per cent and 15 per cent of inhaled cobalt recovered in the urine of the rats and dogs respectively. If one assumes a lung deposition of 50 per cent then 24 per cent of the amount deposited is excreted in the urine in the rat and 30 per cent in the dog.

Rats received a series of single 30-minute exposures to $HCo(CO)_4$; cobalt was determined in the lungs of animals sacrificed 24 hours after exposure and sacrificed two weeks after exposure. There were also determinations on rats which had received three months of 6 hr/day, 5 days/wk exposure. These were run at the end of the last exposure, 24 hours later, and three months later.

The results of the acute runs are shown in Figure 9. The ordinate shows the quantity of cobalt in milligrams found in the lungs per rat. The abscissa is labeled in two ways, (a) the chamber concentration during the exposure in

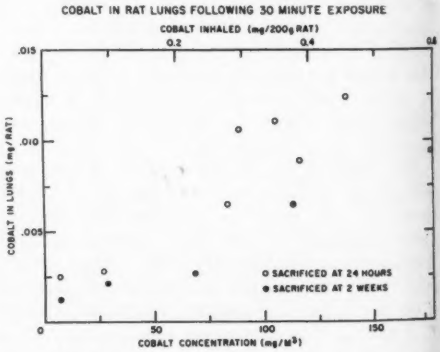
COBALT IN RAT LUNGS FOLLOWING 30 MINUTE EXPOSURE
COBALT INHALED (mg/200g RAT)
COBALT IN LUNGS (mg/RAT)
○ SACRIFICED AT 24 HOURS
● SACRIFICED AT 2 WEEKS
COBALT CONCENTRATION (mg/M³)

FIGURE 9. Cobalt in rat lungs following a series of 30-minute exposures to cobalt hydrocarbonyl.

mg/M³, and (b) the quantity of Co inhaled by a 200 g rat (considered representative) during the 30-minute exposure. Each value is the average of four or five individual lung determinations. The largest number of analyses was made on animals sacrificed at 24 hours; here there is a general trend of higher values at higher concentrations. However, the quantities of cobalt found in the lungs appear to be a small fraction of that inhaled. Typical estimates are about 2–3 per cent of the amount inhaled or 4–6 per cent of that deposited. It is apparent that the preponderant fraction of inhaled cobalt is not stored for even 24 hours in the lung.

The two-week values are considered to show only a small drop from the 24-hour values. It was noted in the discussion of the urinary cobalt values above that the urine also showed low levels for two weeks after cessation of exposure with both rats and dogs.

Analyses of the lungs of six rats sacrificed immediately after coming out of the chamber at the end of the repeated exposures showed an average of 0.031 mg Co/rat, six other animals from the same group sacrificed 24 hours later had lung contents of 0.023 mg Co. Considering that these rats inhaled an estimated 0.47 mg during a single 6-hour exposure, the retention would be about 6 per cent of the inhaled cobalt immediately after exposure and 5 per cent after 24 hours; these retentions are estimated to be equivalent to 14 and 10 per cent of deposited cobalt. Another group of nine rats was sacrificed three months later. Of these, six were negative (less than 6 μg), one had 6 μg and two others had 9 μg Co. Thus some animals can retain small but measurable amounts of Co in their lungs for quite a considerable period.

The results for cobalt distribution for animals on the repeated exposure are summarized in Table V.

TABLE V

Cobalt Distribution Following Inhalation Exposure 6 hrs/day, 9.0 mg Co/M.³

	Dog		Rat	
	Mean	Std. Dev.	Mean	Std. Dev.
Urine—mg Co/day	0.79	0.30	0.064	0.015
Urine—mg Co/l.	4.3	1.1	—	—
Inhaled—mg Co/exposure	5.1	0.6	0.52	0.12
Excreted/Inhaled	0.15	0.06	0.12	0.04
Blood*—mg Co/100 g	—	—	0.012	—
Lung*—mg Co/lung	—	—	0.032	—

* Rats sacrificed immediately after exposure.

Pathology

Gross pathological examination of the animals was done on both those acutely and chronically exposed. All tissues were fixed in 10 per cent formalin. Hemotoxylin-eosin stain was used routinely; special stains were employed where indicated.

Pathological evaluation was made on controls and on animals given cobalt hydrocarbonyl. Animals were sacrificed at either 24 hours or at two weeks after exposure.

Characteristic gross and microscopic changes were found in the lungs of those animals which were sacrificed 24 hours after the exposure. Microscopically in almost all animals exposed to cobalt at the higher carbonyl levels there was a striking congestion of the lungs characterized by a deep red color comparable to the color of the liver. The lungs were firmer than usual and edema could be easily demonstrated by expressing frothy pink fluid from the cross cut bronchi.

The lungs which showed characteristic gross changes did not show intra-alveolar edema fluid microscopically. In view of the difficulties in the histological demonstration of edema, the dilatation of the lymph vessels was compared in the various groups along the lines suggested by Drinker[10] for the evaluation of edema. The lymphangiectasia was found to be parallel with the change of lung/body weight ratios discussed above; it was characteristic of rats exposed to higher concentrations of cobalt and sacrificed at 24 hours.

While the above changes were characteristically found in animals sacrificed 24 hours after the exposure, they were hardly detectable in animals sacrificed 14 days later. These changes indicate that a short exposure to cobalt hydrocarbonyl in experimental animals results in lung edema, and in a beginning pneumonia which disappears for the most part within two weeks after the exposure.

In the second part of the experiment, animals were exposed to cobalt carbonyl (3, 9 or 71 times) and were sacrificed either immediately, three months or six months after the last exposure. (A group of exposed rats has been set aside for lifetime observation. The findings on this group will be reported later.)

There was found a very characteristic change in the lungs of those animals which were immediately sacrificed after the 71 exposures. Numerous small yellowish-white nodules scattered throughout the lungs and under the pleural surfaces were detectable. Microscopically the

nodules consisted of large macrophages with foamy cytoplasm sometimes containing minute brownish-black particles. These aggregates were found either around the bronchi or under the pleura. There was no surrounding inflammation or fibrotic reaction. In the rats and guinea pigs the nodules were Sudan-negative, while in the dogs they proved to be Sudan-positive. In the center of the nodules large cholesterin-like crystals were easily demonstrated by the use of the polarizing microscope. The above changes were present in both dogs and to a varying extent in the rats and guinea pigs.

Additional changes included a moderate interstitial and peribronchial fibrosis, mild emphysema and a moderate peribronchial lymphoid hyperplasia. It was found that for the production of these changes, i.e., foam cell aggregates, it is necessary to expose animals repeatedly to cobalt inhalation. The foam cell aggregates disappeared with time. Thus the animals sacrificed three or six months after 71 exposures did not appear to be different from the control animals of the same age. The foam cell aggregates were not evident and no sign of subsequent fibrosis was found. The nature of the foam-cell aggregates is not known. It seems reasonable to assume that they represent a reaction to endogenous lipid accumulating as part of the response to the irritant.

Exposed and control animals both showed signs of bronchitis, peribronchial fibrosis, increase in peribronchial lymphoid tissue, and bronchiectasis consistent with rats of this age. All experimental and control guinea-pigs showed parenchymal and peribronchial lymphoid aggregates.

For the examination of a possible allergic sensitization to cobalt a small number of previously exposed animals (five guinea pigs) was exposed to cobalt inhalation after 71 exposures and a six-month post-exposure interval. Two of three guinea pigs sacrificed at 24 hours after exposure revealed eosinophilia in the lungs. One of the two demonstrated a definite eosinophilic pneumonia, not observed in any other control or experimental animal.

The following organs were examined and found to be without significant changes in exposed and control animals: heart, kidney, spleen, liver, stomach, intestine, adrenals, thyroid, testis, lymph nodes, bone marrow, and brain.

Changes found in the lungs of animals exposed once to cobalt inhalation may be summarized as follows: (a) gross edema, (b) elevated ratio of lung/body weight (c) lymphangiectasia, (d) early pneumonia. Immediately after 71 exposures the presence of foam cell aggregates was found to be characteristic. After 71 exposures and a longer suvival time, the usual observations were: (a) the disappearance of the foam cell aggregates, (b) lack of fibrosis, and (c) bronchial changes characteristic of age.

Discussion

It is clearly apparent that cobalt hydrocarbonyl rapidly decomposes in air. Virtually all of the cobalt in the argon delivery stream is gaseous; however, even in the rapidly ventilated chamber used in the acute runs, less than 10 per cent of the cobalt was found in the gaseous form. In more slowly ventilated chambers used for the repeated exposure, a negligible amount of gaseous cobalt (less than 1 per cent) was found. For field use particulate collection is essential and, in fact, may be of dominant importance. Thus this work suggests that the toxicity observed here was not from the hydrocarbonyl but probably from simple inorganic cobalt compounds. Comparison of our results with available data on the toxicity of cobalt oxide shows some support for this point. The data secured by Carpenter[8] would indicate that a relatively short series of repeated 6 days/wk, 8 hrs/day exposures at 50 mg/M^3 of CoO, was fatal for most of the rats tested; this is equivalent to 39 mg/M^3 of Co, a value about ⅕ the 30-minute LC_{50} as determined here. A comparison of Carpenter's results with those of the repeated runs reported here shows that about five times as high cobalt concentration as that used in these studies (39 mgm/M^3 compared to 9 mgm/M^3) produced much more pronounced toxic effects.

The acute studies as a whole indicated that the cobalt hydrocarbonyl under the conditions employed was a strong pulmonary irritant. The chemical and pathological picture following hydrocarbonyl exposure is consistent with that of an acute chemical pneumonitis. The effects are comparable to those observed after nickel carbonyl exposure as reported by Kincaid et al.[11] and as carried out here. The 30-minute $Ni(CO)_4$ LC_{50} reported for rats is about 85 mg Ni/M^3 (240 mg $Ni(CO)_4$/M^3). This compares with 165 mg Co/M^3 as the corresponding LC_{50} for cobalt hydrocarbonyl. (A series of 30-minute exposures to $Ni(CO)_4$ has been completed in our laboratories; these experiments will be reported later. The results are similar to those reported by Kincaid et al.[11])

Repeated exposure to cobalt hydrocarbonyl revealed both acute and long-term effects of this material. There were deaths in the rats on all the repeated runs, but these did not appear to

be related to the length of exposure. In fact, it was seen that most of the mortality on the 71 exposure experiment occurred during the first week of exposure. For this reason, if dose related, the deaths are regarded as acute rather than chronic effects. It was not possible to rule out acclimatization to the regimen itself as a contributing factor, nonetheless, the mortality rate did appear excessive during the first weeks of exposure.

There is apparently little holdup of cobalt after inhalation, so that urinary concentration drops rapidly (50 per cent in one day) after termination of exposure.

The finding of increased hemoglobin was directly related to exposure but was not, however, necessarily a deleterious effect.

The appearance of nodules grossly and of foam cells microscopically in the repeatedly exposed dogs was a striking effect, but its significance is not known. The lungs of the two dogs sacrificed two weeks after exposure ended certainly did not appear to be normal. The other two were sacrificed six months after the last exposure and appeared normal. This indicated that the foam cells disappeared after removal from exposure and did not leave any residue.

Of three of the repeatedly exposed guinea pigs given a single additional exposure six months later and sacrificed 24 hours after that, one showed an eosinophilic pneumonia. This was the only time this was seen in any test or control animal; it is considered to be suggestive of an allergic response.

Summary

Cobalt hydrocarbonyl was submitted to toxicological examination. A study of the stability of the hydrocarbonyl in air and in other gases was carried out; cobalt hydrocarbonyl was shown to decompose rapidly in air and to yield a dust, probably of inorganic cobalt compound(s). Methods were developed for sampling, analysis and characterization of cobalt hydrocarbonyl and its decomposition products.

The LC_{50} for rats for a single 30-minute exposure was approximately 165 mg Co/M³. The acute clinical signs as well as gross and microscopic pathology indicated a chemical pneumonitis. Available data show a marked similarity of acute effects between $HCo(CO)_4$ and $Ni(CO)_4$; on the basis of 30-minute rat LC_{50}'s the cobalt compound is about one-half as potent as $Ni(CO)_4$.

A three-month repeated exposure to cobalt hydrocarbonyl decomposition products was conducted using rats, guinea pigs, and dogs; cobalt concentration in air averaged 9 mg/M³ for the 71 six-hour exposures. Toxicity was evaluated in terms of clinical condition, biochemistry, hematology, and gross and microscopic pathology. Except for some early mortality, repeatedly exposed animals appeared to be in good physical condition.

Chemical analyses of lungs and urine of exposed animals were carried out to determine the kinetics of the metabolic handling of inhaled cobalt. It was found that there is a rapid turnover of the bulk of the inhaled material, a substantial fraction being found in the urine within one day after exposure; the urinary level drops by roughly one-half for each day after removal from exposure. A definite and persistent elevation in rat hemoglobin values was observed; this is interpreted as a physiological change due to cobalt.

Animals were sacrificed immediately after and at three and six months after termination of exposures. The most noteworthy pathological change was the production of foam cell aggregates in the lungs of repeatedly exposed animals; these disappeared after termination of exposure. There was no permanent damage demonstrable by the techniques employed.

Acknowledgments

The authors wish to thank Dr. Milton Orchin of the University of Cincinnati for supplying the first pyridinium cobalt carbonyl and for his helpful comments during the course of the work. The above material was later supplied by Dr. Vincent L. Hughes of Chemical Research Division of Esso Research and Engineering Company; this is gratefully acknowledged. The authors wish to thank Mr. M. Talvitie of the Occupational Health Program, USPHS, Cincinnati, for carrying out x-ray diffraction studies on the cobalt hydrocarbonyl decomposition products. Appreciation is expressed to the Institute of Industrial Medicine Staff who participated in one or more phases of the study: Lester Barrer, M.S., Cecile A. Broadhurst, E. Christofano, B.S., John Donovan, M.P.H., Krystyma Gordon, B.S., Jamil Haddad, M.D., Marcella Mason, and John Molnar, M.D.

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Translation of Russian Air Pollution Study

A TRANSLATION BY Dr. B. S. Levine, U.S. Public Health Service research grantee, of the Russian book *Sanitary Protection of Atmospheric Air: Purification of Industrial Discharge Gases from Suspended Substances* has been published for sale to the public by the Office of Technical Services, U.S. Department of Commerce. The book was written by V. N. Uzhov of the Technological Division of the Institute for Making Gas Purifying Equipment in Moscow. The translation (item 59-21092) may be ordered by title from OTS, U.S. Department of Commerce, Washington 25, D.C., price $3.

Included in the 160-page volume are chapters on "Properties of Gas Suspensions," "Methods of Mechanical Gas Purification," "Wet Gas Purification," "Gas Filtration," "Electrical Gas Purification," "New Methods and Apparatus for Gas Purification," "Combination Gas Purifying Apparatus," "Flue Gases from Heat and Power Stations and Boiler Plants," "Elimination of Ash from Flue Gases from Large Electric Power and Heat Centers," "Removal of Ash from Boiler Plant Flue Gases," "Purification of Emission Gases in Plants of Ferrous Metallurgy," "Purification of Discharge Gases of Non-Ferrous Metallurgical Plants," "Purification of Gases Discharged by Cement Plants," "Purification of Gases Discharged by the Chemical Industry," and "Purification of Ventilation Air from Dust."

Dr. Levine's translation of the three-part Russian book *Limits of Allowable Concentrations of Atmospheric Pollutants,* edited by Prof. V. A. Ryazanov in Moscow, has been printed and is also available from OTS. The three volumes, all of which appear under the same title, are: 59-21173 Book 1, 1952, 145 pages, $2.75; 59-21174 Book 2, 1955, 179 pages, $3; and 59-21175 Book 3, 1957, 152 pages, $3.

Diethylstilbestrol Absorption in Industry: A Test for Early Detection as an Aid in Prevention

R. M. WATROUS, M.D., and R. T. OLSEN, B.S.

Employee Health Department and Control Laboratories, Abbott Laboratories, North Chicago, Illinois

TWO YEARS after the discovery by Dodds of diethylstilbestrol, Dunn[1] described the effect of the drug on a man who received 5 mg per day for 75 days. There was enlargement of the breasts, loss of sexual drive and a decrease in the size of the external genitalia. In 1941, Scarff and Smith[2] reported what appear to be the first cases of diethylstilbestrol absorption in chemical workers. They examined specimens of hypertrophied breast tissue from two young men who had been handling diethylstilbestrol and its precursors for ten and twelve weeks, respectively. The breasts had enlarged three to four weeks after the beginning of this work. The histological sections showed "a marked degree of epithelial overgrowth in the acini and ducts, with, in places, solid acinar formation, but no definite invasion of interstitial tissue by epithelial cells. There was a moderate degree of new fibrous tissue formation, but little retention of secretion."

In 1944 Fitzsimons[3] published her classical description of diethylstilbestrol absorption in 20 chemical workers, and described the elaborate measures required to prevent this. The earliest symptom reported by these workers was a tingling sensation in the nipples, appearing usually about three weeks after exposure began, but occasionally felt as early as the fourth day. On continued exposure, a small area of thickening could be palpated in the breast, usually under the areola. These changes regressed slowly after exposure to the chemical ceased. To prevent or minimize exposure in this factory, it was necessary for the workers to wear full cover-alls, dust masks and rubber gloves. The operation had to be isolated, with entry accomplished through an air-lock and shower. Within the room itself, extreme care had to be taken against spills or dusting. Ovariectomized mice were often placed in cages inside the room, and if contamination of the air occurred estrous changes were readily demonstrated in smears from their vaginas.

In 1949, Goldzieher and Goldzieher[4] reported another industrial case in a worker who extracted natural estrogens from pregnancy urine. After six months of this work, the man complained of enlarged breasts, lessened sex drive and a decrease in the size of the penis. Assay of the urine for estrogens showed an excretion of 185 ± 20 micrograms in 24 hours. By the same test, the urine of unexposed men showed excretion of 6 micrograms in the same period. After the patient had been removed from his work for a month, his excretion had decreased to 11 ± 2 micrograms per 24 hours.

In 1950, Fisk[5] reported five cases of enlarged breasts in workers handling conjugated natural estrogens in a chemical plant in Canada. Assay of the urine showed high values in each case; these ranged from 298 to 400 micrograms per 24 hours, values stated to be ten times normal by the test used.

Pagani[6] reported a number of cases of diethylstilbestrol and other synthetic estrogen absorption in both male and female workers in Italy. The women complained of menorrhagia, and were found to have endometrial hyperplasia. The men complained of the usual itching and sensitivity of the nipples, pigmentation of the areola, dilation of areolar veins, subareolar masses, loss of sex drive, poor erections, diminished volume of semen, and edema of the scrotum (two cases). One case of home contamination was reported in this series; the prepubertal daughter of a night worker slept in his bed while he was at work, and developed *pubertas praecox*.

Klavis[7] reported several cases in a German pharmaceutical factory, while Katzenellenbogen[8] and Klofrajn and Bartini[9] reported cases in Israel arising from the manufacture and use of diethylstilbestrol pellets for caponizing cockerels. Several children who had home contact with these workers developed pigmentation of the nipples and linea alba. Measurements of estrogen excretion were made on the urine of four of the people involved in this incident. Two men showed 20 to 70 Allen-Doisy mouse units per liter; one woman showed 400 and 300 units at different times; a boy showed 10 units. If one mouse unit is taken as 0.02 micrograms of diethylstilbestrol,

these figures would seem unusually low: 0.4, 1.4, 8.6, and 0.2 μg per liter.

A recent case of home contact with diethylstilbestrol in connection with poultry raising is reported by Green.[10] Two young children and the wife of a poultry farmer were affected.

A curious source of contact with diethylstilbestrol is reported from Holland, where children developed pigmentation and gynecomastia from the use of a hair tonic which contained the chemical.[11]

Personal Experience

In 1956, production of diethylstilbestrol on a large scale was planned at Abbott Laboratories. Through correspondence with K. C. Kohlstaedt, of Eli Lilly and Co., we learned of a biological test performed on the urine of workers at weekly intervals to detect any faults in manufacturing technique which might lead to absorption. This led us to develop a somewhat different test, based on the method of Lauson *et al.*[12]

In this test, immature female mice of the CF-1 strain weighing eight to ten gm are injected orally once each day for three days with 0.2 ml of diethylstilbestrol standard or urine to be tested. Two levels of standard are used: 0.06 and 0.18 μg/ml respectively. Groups of ten mice are used for each level of standard and for the urine. The urine is fed undiluted and untreated. On the fourth day the mice are killed with chloroform. The uteri are separated from the vaginas by cutting through the cervix. The surrounding tissue is stripped off and the utero-tubal junction is cut. The uteri are placed in a closed petri dish with a piece of wet filter paper in the cover until they are weighed. Just before weighing, intra-uterine fluid is pressed out between two pieces of Whatman No. 1 filter paper. The uteri are weighed in groups of ten on an analytical balance to the nearest 0.1 mg. Uteri from untreated control mice average about 8 mg each; those from mice receiving 0.06 mcgs of diethylstilbestrol per ml average 16 mg each; those from mice receiving 0.18 mcgs of diethylstilbestrol per ml average about 31 mg each.

Intermediate Compounds

In the synthesis of diethylstilbestrol several intermediate compounds had to be handled, and it semed desirable to test their estrogenic effect, if any. In this way we could determine whether to extend stringent precautions to the intermediate steps or to confine them to the final product. The compounds were tested, and their estrogenic effects were expressed as fractions of that of diethylstilbestrol. The activities ranged from 1/320 to less than 1/1000 that of the pure substance. In view of these results, no special precautions were taken in handling these intermediates; this was later justified by the fact that workers in this part of the process never showed any estrogenic activity in their urine, or any symptoms of the estrogenic type.

Exposure to pure diethylstilbestrol occurred in the final stages of synthesis, in which the material was recrystallized, filtered in a basket centrifuge, dug out with a hand scoop and transferred to a closed blender-dryer. The dried material was in some cases passed through a mill to the final container, and in some cases transferred directly without milling.

These operations were all segregated in one room, which had tile walls free of ledges to facilitate washing down. Workers entering the area passed through an anteroom where their street clothes were stored along with clean workclothes. The workers stripped completely and put on underwear, socks, rubber boots, coveralls with ties at the bottom of the trousers for tying over the boots, rubber gloves, cloth caps, and dust masks of the A-O type. They then proceeded past a shower area into the main work room. The room was ventilated by an exhaust fan discharging through a bag filter. The exhaust from the blender-dryer took place through a water aspirator directly to the sewer. The room contained a washing machine for the clothes and facilities for decontaminating the boots and gloves. An alkaline detergent, "Pennsal Regular," was found most suitable for washing the clothes and wiping surfaces.

The workers left the room after discarding all used clothing and passing through the shower to the clean clothes area.

In spite of this arrangement, the chemical workers first assigned to this job usually showed increased diethylstilbestrol excretion within the first one or two weeks. It was necessary to appoint as supervisor a man who had previously been in charge of handling sterile bulk powders under aseptic conditions. His standards of cleanliness and knowledge of the fine points of asepsis soon brought out many small ways in which the material being handled was becoming disseminated into the environment. When refined procedures were explained to the chemical operators, this dissemination was reduced, and a particularly careful and cooperative worker could do the job for many weeks without showing appreciable increase in excretion. Not every worker, however, could or would observe the precise technique required, and we cannot say that the problem was more than barely solved by all the methods we could devise.

Urinary Excretion

The urine samples tested were passed in the morning during the latter part of the week during which the operator had worked with pure diethylstilbestrol, and the tests were performed at weekly intervals. The values were expressed in decimal fractions of a microgram per ml of urine. Values below 0.02 microgram (the lower limit of sensitivity) were logged as zero, and these were consistently observed in workers not exposed to the final product. It was found convenient to plot these values from week to week as a simple graph. Figure 1 shows such graphs for five workers who were in and out of the process during the last half of 1956.

Case 1: Showed urinary levels ranging between 0 and 0.1 micrograms per ml from the start. At point A on the graph he complained of tenderness of the nipples and was removed from the process.

Case 2: Showed rising levels of excretion for the first three weeks. At point A on the graph he was interviewed, but showed no objective signs of estrogenic effect; he had no complaints at this time. At point B, he complained of tenderness in the left nipple, and some induration was present. He was removed from the job, but the objective changes in the breast were still present at point C.

Case 3: Is typical of workers who handled precursor chemicals only and were not exposed to the final product.

Case 4: Showed very high levels of excretion on two occasions at a time when the process was just getting under way. He was not seen in the Medical Department, and made no complaints, but when questioned later he recalled he had noticed some tenderness of the nipples without enlargement.

Case 5: After working about three months with precursor chemicals, this man began to handle finished diethylstilbestrol. He demonstrated increasing levels of excretion, up to 0.30 micrograms per ml. At point A on the graph he was examined and found to have subareolar induration. He was removed from the job, and the changes regressed over a period of months.

Figure 2 is a graph of the excretion of a worker who seemed to be relatively insensitive to diethylstilbestrol. His excretion reached a peak of 0.07 μg/ml late in 1957, and at point A he had no objective or subjective evidence of estrogenic effect. After a short exposure, with no evidence of absorption, in the spring of 1958, he began full-time work in diethylstilbestrol in the fall at point B on the graph. In spite of urinary excretion which reached 0.20 μg/ml, questioning and examination at point C revealed no changes.

Figure 3 is the excretion graph of another worker who was intermittently exposed during this same period. At point A he noticed some itching of the nipples and some subareolar induration, yet at point B, where he excreted as much as 0.47 μg/ml, he noticed only slight itching.

From our experience we believe that periodic monitoring of the urine will give evidence of diethylstilbestrol absorption before any marked physical signs appear. Corrections in handling technique can thus be put into effect earlier.

According to studies of excretion following known dosage in human beings[13, 14] diethylstilbestrol begins to appear in the urine immedi-

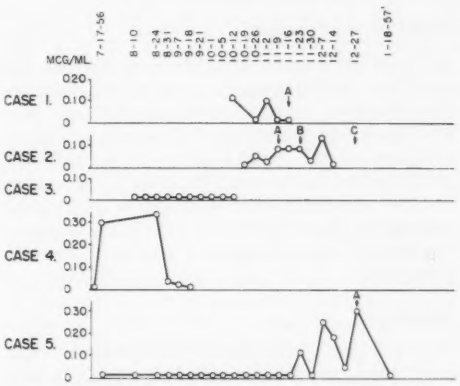

FIGURE 1. Diethylstilbestrol excretion of five workers, 1956.

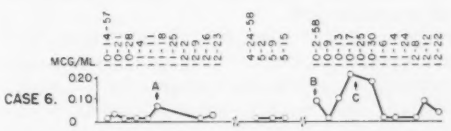

FIGURE 2. Diethylstilbestrol excretion curve for a worker, 1957–58.

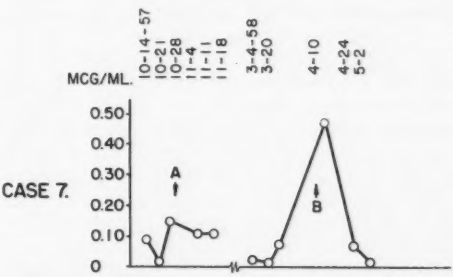

FIGURE 3. Diethylstilbestrol excretion curve for a worker, 1957–58.

ately, and reaches its peak level on the third day after a single dose. Ten to 30 per cent of the dose is excreted in the urine within ten days; the remainder is excreted in the feces. By making a number of assumptions, one may calculate that a man excreting diethylstilbestrol at a level of 0.10 μg/ml is probably absorbing the chemical at the rate of 0.3 to 1.0 milligrams per day. This is not too far out of line with clinical experience in cases where the drug is given by mouth, but is a very rough approximation, and might be on the high side by a factor of ten. Absorption may theoretically be percutaneous, oral or respiratory, but in our cases we believe it was principally through the skin, because of the protection provided for the other routes.

Extent of the Problem

Tariff Commission Reports does not list the amout of diethylstilbestrol manufactured in the United States as a separate item, but our own experience and knowledge of other major manufacturers in this field lead to an estimate of 10,000 pounds or more per year. As an increasing amount is being used in animal husbandry, the number of those exposed must be increasing at a rapid rate. This is confirmed by a review of the literature.

Summary

1. A review of the world literature shows that since 1941 the absorption of diethylstilbestrol by chemical workers has been a problem everywhere. More recently, the use of this chemical in animal husbandry has increased the number of people exposed.

2. A bioassay procedure is described which can be used to measure the urinary estrogen output of diethylstilbestrol workers and provide an index of the amount absorbed. If done weekly, the test detects absorption before clinical signs appear.

3. Unexposed chemical workers excreted estrogens below the threshold of the test (0.02 micrograms per ml of urine). Exposed and imperfectly protected workers showed values ranging from 0.06 to 0.55 μg/ml. Earliest symptoms of breast stimulation were seen one to two weeks after urinary excretion reached or exceeded 0.1 μg/ml.

4. Elaborate precautions required to control exposure are described.

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Experience with the Massachusetts Code on Ionizing Radiation

HAROLD BAVLEY and ROBERT L. DEVLIN

Massachusetts Department of Labor and Industries, Boston, Massachusetts

AS A RESULT of a special staff study, the Commissioner of Labor and Industries, empowered by the Legislature under Chapter 149, Acts of 1919, appointed an advisory committee in February, 1957, to prepare a code for regulating the hazard of ionizing radiation in Massachusetts industry. This committee, representing labor and industry, with the aid of experts well versed in this potentially serious hazard, concentrated on this problem and formulated an acceptable all-inclusive flexible code. Subsequent to a public hearing in July, 1957, the code was adopted and became effective on December 1, 1957. Massachusetts became the sixth state to officially promulgate rules and regulations for the protection of employees exposed to ionizing radiation.

The Rules and Regulations Relative to Ionizing Radiation, known as Industrial Bulletin No. 5, are based on the experience of the Massachusetts Department of Labor and Industries, that of similar state agencies, and the U. S. Atomic Energy Commission. The Department first became acutely aware of the hazard of ionizing radiation in 1940, during the expansion of our military potential. While on a routine check of homework permits, one of our Industrial Inspectors discovered that a subcontractor of an aircraft-instrument manufacturer was providing individuals with radium-base paints for application to instrument dials in their own homes. These unsuspecting workers operated without supervision, without hazard controls, and without any knowledge of the potentially serious hazard resulting from the mishandling of radium-base paints. Fortunately, this homework had not involved many workers, nor had it progressed to any great extent before the operation was prohibited by the Department. These workers had escaped the sad experience of an industry in another state, where numerous fatalities had occurred from improper working procedures during the application of radium-base paint. A conference with experts in the field of radioactivity resulted in the development of safe working procedures which are still adhered to by the dial-painting industry in Massachusetts.

Prior to 1946, investigations made to determine the extent of the hazards of ionizing radiation were hampered by the lack of radiation detection instruments. When these instruments became available to the Department of Labor and Industries, a series of special surveys were inaugurated. The first such survey was requested by the Massachusetts Department of Public Health, which was using mobile x-ray machines in their tuberculosis survey. The hazards pertinent to the use of x-ray shoe-fitting fluoroscopes were uncovered in 1948, during a survey requested by the Boston Better Business Bureau. The use of these machines in Massachusetts was prohibited on February 17, 1958.

During 1950, when shipments of radioisotopes from the Atomic Energy Commission's reactor at Oak Ridge, Tennessee, began to infiltrate Massachusetts, a joint survey with the Department of Public Health was instituted to determine the extent of the problem, the uses of the radioisotopes, and the precautions instituted to safeguard the users. It is of interest to note that of the thirty prime users in this Commonwealth at that time, only three were industrial establishments.

Research and development during the past decade has resulted in a rapid increase in the application of radioisotopes and other sources of ionizing radiation to industrial processes, a considerable improvement in operational techniques, and a greater use of ionizing radiation in research, analytical procedures, and power development. Many applications of ionizing radiation which were held either impracticable or were merely ideas ten years ago are now in daily use. These applications have resulted in considerable savings to industry, improvement of product, and a benefit to the consumer. However, as this expansion was taking place, it became apparent to the Department of Labor and Industries that closer control was needed and that a system of user identification was necessary in order to be certain that the employees involved in such establishments were fully protected and that the hazards resulting from the use of ionizing radiation were satisfactorily controlled. In the past,

this had been accomplished successfully in other fields by promulgation of Rules and Regulations which required registration.

Registration of users of the various sources of ionizing radiation was considered essential if the scope of the problem was to be fully evaluated. Existing installations and sources to be used by an employer outside an installation were required to be registered within ninety days after the effective date of these Rules and Regulations, i.e., December 1, 1957, and every new installation must be registered before it is placed in operation. A "one-time" registration was put into practice and found to be satisfactory unless the change in the installation or source presented an entirely different degree of hazard from that originally registered. The registration forms were completed by the employer in triplicate and forwarded to the Department of Labor and Industries. A copy was numbered, receipted, and returned to the employer for his records. The remaining copies were for the use of the two divisions, the Division of Industrial Safety and the Division of Occupational Hygiene, which cooperate in the enforcement and the provision of technical advice to the registrants. If the employer increased his sources of ionizing radiation, he was requested to supplement his registration by writing a letter to the Department. The acknowledgment of registration does not imply the approval of the Commissioner of such an installation, but merely indicates that the Department has a record of such an installation.

As of June 1, 1959, there are 284 installations registered with the Department of Labor and Industries. Geographically, these registrants are distributed throughout 92 cities and towns within the Commonwealth. Twelve registrants are located in other states but occasionally perform their operations in Massachusetts. The greatest concentrations of registrants are centered in and about the largest cities. A total of 93 industrial registrants are located within the confines of Metropolitan Boston. Approximately 200 registrants, or 70 per cent of the total, are located in the eastern third of the state. This distribution seems to follow the industrial pattern existing at the present time in this Commonwealth.

TABLE I

Registrants by Industry

Instrument mfg.	12	Printing	10
Paper products	34	Chemical	15
Electrical and electronic	40	Nuclear	13
Rubber and plastics	28	Radiography	12
Machinery	16	Entertainment	7
Textiles	13	Food	5
Metal fabrication	45	Insurance	3
Foundry	8	Communication	2

Research laboratories—19

TABLE II

Registrants* and Their Radiation Sources

Type of source	Number of registrants	Type of source	Number of registrants
AEC Radioisotopes	73	Particle accelerators	4
Special gauges	59	Electron microscopes	8
Static eliminators	47	Neutron sources	4
X-ray machines	104	Thorium	34
High voltage equipment	29	Uranium	15
		Radium	22

* Each registraint may use one or more sources.

The registrants represent the basic industries in Massachusetts, with similar distributions found in both heavy and light industry. Table I represents a summary of the registrants classified by industry. The metal trades, fabrication and foundries, have a total of 53 installations. Most of the fabrication shops are engaged in the handling of a magnesium alloy containing 3 per cent thorium. The U. S. Atomic Energy Commission requires the user of this metal to be licensed because it contains thorium, a source material. The amount of alloy licensed in Massachusetts during the past year is approximately 69,000 pounds, amounting to a content of over 2,000 pounds of thorium. The Massachusetts "code" requires registration of this alloy if the registrant possesses more than the equivalent of 4.9 pounds of thorium.

The industrial users of the sources of ionizing radiation in Massachusetts, with certain exemptions, are required to register such sources with the Department of Labor and Industries. The registrants must provide information as to the type of source, i.e., radioactive materials, x-ray machines, or other high voltage equipment. Table II indicates the types of sources and the number of registrants. Some industrial establishments have more than one source of ionizing radiation.

Approximately 50 per cent of the registrants are licensed by the U. S. Atomic Energy Commission to possess radioisotopes for use in special operations or in instruments, such as thickness gauges. A list of the most popular radioisotopes used by industry is found in Table III. Radium, used by 72 registrants, appears to be the most prevalent. Radium is primarily found in static eliminators, special vacuum gauges, and is used in the dial painting industry.

TABLE III

Most Popular Radioisotopes in Massachusetts
(Used by 12 or more registrants*)

Radioisotope	Number of registrants
$Cesium^{137}$	20
$Cobalt^{60}$	39
$Iridium^{192}$	16
$Krypton^{85}$	15
$Radium^{226}$	72
$Strontium^{90}$	48
$Thallium^{204}$	13
$Thorium^{232}$	34
$Uranium^{238}$	15

* Registrants may use more than one type of radioisotope.

TABLE IV

Equipment Registered as Sources of Ionizing Radiation

Equipment	Units	Equipment	Units
X-ray machines	217	Static eliminators	958
High voltage equipment	142*	Particle accelerators	4*
Electron microscopes	8*	Thickness gages	93*
Vacuum gages	44*	Reference sources	76*

* Does not include manufacturers of equipment.

This material is the only radioisotope that is not under the jurisdiction of the U. S. Atomic Energy Commission. However, the Massachusetts Department of Labor and Industries, since 1940, has exerted rigid controls over this potentially hazardous material. The industrial establishments using radium have co-operated satisfactorily in establishing safe working practices which were developed by experts in the field of radioactivity during a special conference. The incidence of injury in Massachusetts due to this material has been negligible to date. $Strontium^{90}$ is used to a great extent in special gauges requiring, at present, an AEC license. There are a total of 93 gauges registered with the Department. $Strontium^{90}$ has also been used in the manufacture of eye applicators for the medical profession and is currently being used in radiochemistry.

The importance of equipment as sources of ionizing radiation is shown in Table IV. Some 1,542 units have been registered. Radioactive static eliminators still dominate this field. These static eliminators vary from 1 inch to 10 feet in length. Usually each linear inch contains 50 microcuries of radium. Industrial x-ray equipment is the second most prevalent. These machines are rated as to kilovoltage of the x-ray tube. There are 113 x-ray units rated below 100 kv, 100 units rated at 100 to 300 kv, and four units operated in the million volt range. Over 70 per cent of the x-ray units are used in nondestructive testing, 50 per cent for radiography, and 20 per cent for fluoroscopy.

More than 40 different radioisotopes are being used currently by industry in Massachusetts in amounts varying from microcurie to multicurie sources. Table V lists various isotopes used in millicurie amounts; Table VI lists those isotopes used in curie amounts. The total activity of all radioisotopes currently registered in this Commonwealth exceeds 28,000 curies. This factor is indicative of the rapid increase in the quantity of radioactive material used by industry from the initial start of a few curies first used in 1950.

Since it is not the intent of the Department to harass licensees of the U. S. Atomic Energy Commission who are required to abide by the regulations of the Atomic Energy Commission, the Rules and Regulations as adopted by the Massachusetts Department of Labor and Industries are intended to be in harmony with the Federal regulations as they apply. Industry and labor have been co-operating with the Department during the past decade towards the protection of workers exposed to ionizing radiation. Hence, these Rules and Regulations are believed to be reasonable and have worked no undue hardship on either the employer or employee during their

TABLE V

Registered Quantities* of Radioisotopes

Radioisotope	Millicuries	Radioisotope	Millicuries
$Cesium^{144}$	10	$Promethium^{147}$	130
$Chromium^{51}$	15	$Ruthenium^{106}$	40
$Iron^{59}$	73	$Thallium^{204}$	604
$Nickel^{63}$	42	$Tantalum^{182}$	5

* Also microcurie sources not included.

TABLE VI

Registered Quantities of Radioisotopes

Radioisotope	Curies	Radioisotope	Curies
$Carbon^{14}$	4.10	$Strontium^{90}$	64.32
$Cesium^{137}$	20.51	$Sulfur^{35}$	2.50
$Cobalt^{60}$	2047.60	Thulium	500.00
$Iodine^{131}$	2.00	Thorium	10.00
$Iridium^{192}$	17437.00	$Radium^{226}$	5.44
$Krypton^{85}$	142.44	$Uranium^{238}$ and $Uranium^{235}$	100.00
$Phosphorus^{32}$	2.10		
$Polonium^{210}$	1.08	Tritium	8024.15

proper enforcement. Since the promulgation of the "code", 310 visits were made to 195 plants. In addition, many pre-operational visits, telephone calls, and scrutiny of plans were completed during this period as a consulting service to plant management on their contemplated problems and work procedures. At 104 of these plants, recommendations were made to bring the installation into compliance with the Rules and Regulations. Most of these establishments were not acquainted with the "code" requirements necessitating additional educational work on the part of the Department's representatives. Nearly all of the recommendations were completed within a short time by merely directing the attention of the employer to the recommendations by a letter. Only six written Department orders have been issued during this period. No court prosecutions have been necessary to date. In every instance, there has been evident a spirit of friendly co-operation which is not always found present in other hazard fields.

Since the promulgation of the *Rules and Regulations on Ionizing Radiation*, an effort has been made by the Department of Labor and Industries to afford proper coverage of this hazard by establishing an adequate inspection service. Prior to the adoption of these Rules and Regulations, the Division of Occupational Hygiene performed all such technical service for the Department. Plants handling radium or radioisotopes and several installations using x-ray equipment have been visited since 1946, routinely or on special request, by the Division's engineering staff, for evaluating the radiation hazard and recommending proper control measures deemed necessary. However, after the adoption of the "code", it became obvious that this inspection service would require supplementation since the engineering staff handles many problems involving other health hazards in addition to the hazard of ionizing radiation. Accordingly, the full-time position of Industrial Radiation Control Supervisor was created in the Department. The duties include education, evaluation, and enforcement. It is anticipated that all registered installations will be surveyed and brought into compliance with the "code" requirements before the end of this year.

Several thousand employees in Massachusetts are currently working at operations which offer direct or indirect exposure to ionizing radiation. The size of the employee force continues to expand as new installations are registered. Nevertheless, the accident experience from exposure to this hazard since the code's inception has been quite favorable. There has been a total of five overexposure accidents reported to date. Three of these accidents occurred at three different plant locations as a result of the malfunctioning of a mechanically operated shield on an x-ray spectrograph. In each case, first and second degree burns developed on the exposed portions of the employee's hands approximately two weeks after the actual exposure. The company manufacturing the x-ray spectrograph was contacted and advised of the cause of the accidents. Necessary action was taken by this company to remedy the condition causing the accidents. Each employee was examined by his plant physician. Blood tests were taken over a period of several weeks but no abnormal changes were detected. Two overexposure incidents were reported involving the handling of large sources of radioactive material. Each employee was examined immediately after the alleged overexposure. In neither case was evidence of physical damage found, although the employees were checked by their plant physician as well as by the Department's Occupational Hygiene Physician over a period of several months. In all of these overexposure incidents, technical surveys were performed by staff members of the Department to determine the cause of each accident and to recommend the necessary precautions to be taken to prevent the recurrence of such accidents. A total of two days lost time was involved in all of these cases. The accident experience to date has resulted in frequency and severity rates which are much lower than the average for industry in Massachusetts.

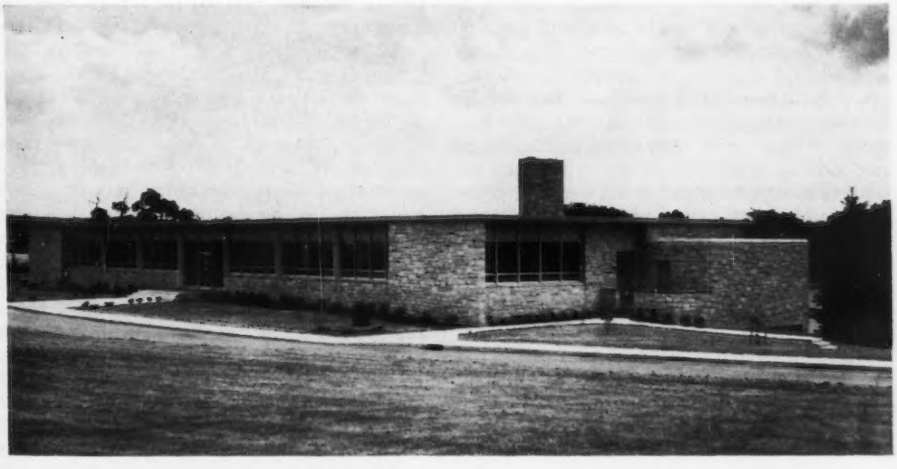
To implement the educational program offered by the Department of Labor and Industries for the control of the ionizing radiation hazard, a series of bulletins has been published by the Division of Occupational Hygiene, covering the most common radioisotopes handled by Massachusetts industry. These bulletins specifically describe the hazards and controls for the individual radioisotopes, and are in addition to the general bulletins on ionizing radiation previously published. As a result of numerous requests by plant physicians for a definition of "appropriate medical examination," as contained in Rules 11.1 to 11.3 of Industrial Bulletin No. 5, the Massachusetts Medical Society, Committee on Industrial Health, was requested to advise the Department as to specific details to be covered by the plant physician in appropriate medical examinations. As a result of the statement issued by the Massachusetts Medical Society, a bulletin, *Medical Examination of Personnel Exposed to Ionizing Radiation*, has been prepared by the Division of Occupational Hygiene for distribution on request.

The present "code" was originally designed to

be all-inclusive in order to extend proper control over all sources of ionizing radiation. However, sufficient flexibility was incorporated in these Rules and Regulations to permit necessary modification in exemptions, maximum permissible doses, and exempt possession limits, whenever it was deemed necessary to conform to the best practice and new information made known through continuing research and experience. During the period in which the code has been in effect, only one company has applied for an exemption from the Rules and Regulations.

The ready acceptance by management and labor of this "code" has permitted effective administration of the "code" by the Department of Labor and Industries. Industry's ready compliance with recommendations, the low accident experience, the single request for exemption, the lack of court prosecutions, the negligible frequency of written orders, are indicative of its proper functioning and suitability for the control of the hazard of ionizing radiation as it exists at the present time in Massachusetts industry. It is the intent of the Department to acquaint industry with contemplated changes so that the future experience with the Massachusetts "code" on ionizing radiation will continue to assure the co-operation presently extended by both labor and management, and result in a continued future favorable experience.

BUSHY RUN CHEMICAL HYGIENE LABORATORY



LATE IN SEPTEMBER, Mellon Institute formally opened the 30,000 square foot, two-story Bushy Run Chemical Hygiene Laboratory, in the country 22 miles east of the Pittsburgh laboratories of the Institute. This building is entirely devoted to the experimental toxicology which Dr. Henry F. Smyth, Jr., and his colleagues have done since 1937 for Union Carbide Corporation. About one-fourth of the work of this group is the obtaining of information required by industrial hygienists and others to produce and utilize new chemicals safely; the balance of the work is devoted to the safety of the consumer.

Health Physics Problems Following a Reactor Accident*

J. M. WHITE, B.A.Sc.

Radiation Hazard Control Branch, Atomic Energy of Canada, Chalk River, Ontario, Canada

AT THE main plant of Atomic Energy of Canada Limited near Chalk River, Ontario, there are two large research reactors, NRX and NRU (Figure 1). The NRU reactor has a heat output of 200 megawatts and is both moderated and cooled with heavy water.

The natural uranium fuel rods are removed from the NRU reactor by raising the rods vertically through the top of the reactor into a rod removal flask. Many safeguards are built into the rod removal flask to prevent the rods from falling out and creating a hazardous condition. An accident of this type is unlikely and only an unusual sequence of events led to its occurrence.

In May 1958, during a normal startup of NRU, the aluminum sheath of one of the fuel rods ruptured. The rod appeared to be badly damaged and attempts to remove it indicated that normal procedures could not be used. It was subsequently removed from the reactor but, because it was badly damaged, cooling water could not be circulated on the rod during removal. The rod overheated, melted and broke into several pieces. One section was later found on the bottom of the reactor vessel; a small amount of smouldering uranium oxide fell on the reactor deck plate around the rod removal port. When the flask was moved, a three-foot section of rod fell into the nearby flask maintenance pit and ignited. A short section of the rod remained in the rod removal flask. Immediately after removal of the ruptured rod from the reactor, when the burning section was noticed, sand was thrown over it to put out the fire. (Figure 2.)

The fields encountered were very high. (Figure 3.) In the vicinity of the flask maintenance pit, fields in excess of 1000R/hr were measured. No measurements were made in the flask maintenance pit but estimated fields were in the range of 10–50,000 R/hr. Fields up to 1000 R/hr were found on the top of the reactor deck plate.

It was estimated that the burned portion of the rod contained 2×10^6 curies of mixed fission products, 700 curies of I^{131} and 13 grams of Pu^{239}. Heavy contamination occurred over a wide area of the reactor hall, offices, change rooms, control room, and rod storage areas. Smear or swipe tests of loose contamination frequently indicated up to 200–300 mR/hr.

Employees present in the building were quickly evacuated through a covered walk-way to the nearby NRX change area. The contaminated men were cleaned up and, if isolated spots of fixed contamination on their person did not exceed 10,000 cpm (counts per minute), they were allowed to go home. The few employees found to be contaminated above this level were sent to the plant hospital for further treatment. There were no cases of severe skin contamination.

Shortly after the accident occurred a crew began checking the area outside the NRU building for air contamination and fallout. Roads and buildings contaminated by fallout were posted and entry was prohibited. With the exception of one or two buildings downwind from the reactor, contamination originating from the accident was slight. (Figure 4.) Decontamination personnel were put to work immediately and within a few hours important roads and buildings were back in operation. During the following week the remaining contamination in less important areas outside the reactor hall was cleaned up. It is of interest to note that more contamination was spread on roads in transporting active material for disposal than by fallout. The roads were cleaned quickly and easily by vacuum cleaners and fire hoses and, when necessary, by removing a portion of the road surface.

After the fire was quenched and personnel evacuated from the buildings, preliminary plans were made for decontaminating the reactor building. It was realized almost immediately that there were not enough men in the reactor crews to handle this job because the extremely high radiation fields would permit only short working periods. The decision was then made to use personnel normally not exposed to radiation and to allow each to receive three Rem.

The first project was removal of the burned

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, April 30, 1959.

out rod sections from the flask maintenance pit and from the top of the reactor. This was done the morning after the fire by means of remote-handling techniques involving the use of the NRU permanent crane and long-handled tools. The work carried out during the following four days was mainly concerned with preventing the spread of contamination and planning for general decontamination of the buildings. It soon became obvious that, if plant employees only were used for the whole job, their permitted radiation exposures would be used up and they would not be available for other active work during the remainder of the year. Outside help was therefore obtained from the Armed Services

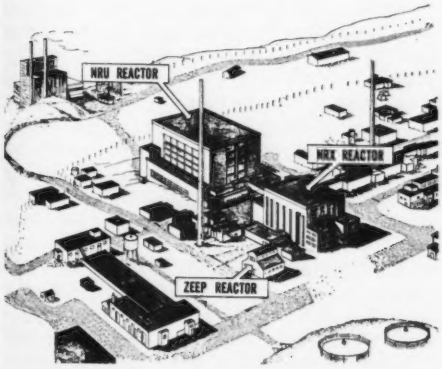
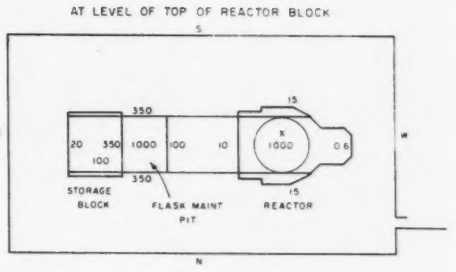
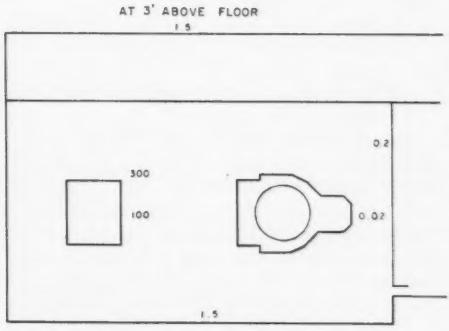


FIGURE 1. View of NRX and NRU.

FIGURE 2. View of NRU showing the rod removal flask, maintenance pit and the top of the reactor.





All numbers are Roentgens/hour

FIGURE 3. Radiation measurements in NRU reactor hall, May 24, 1958.

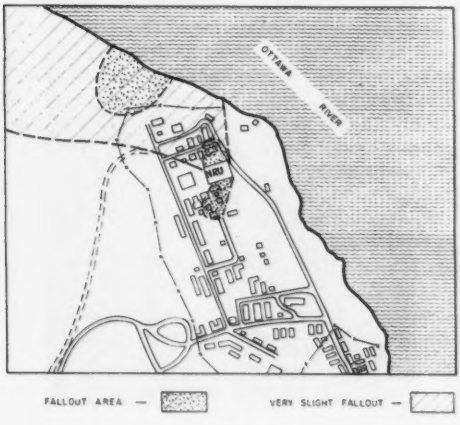


FIGURE 4. Areas contaminated by fallout.

and from the Civil Defence Organization, and lectures on the control of radiation hazards were arranged as part of the program.

Before entering the reactor building all personnel were briefed on the work they were expected to do. The briefings were carried out by reactor personnel and included a view of the work area by means of closed-circuit television. Persons trained in health physics were in attendance during the briefings to emphasize working times and the importance of health physics techniques, and to explain the significance of exposures.

Decontamination of the building was divided roughly into three phases:

(1) Removal of rod fragments and debris.
(2) Removal of contaminated sand and "hot" spots.
(3) Removal of contamination from walls, floors, fixed equipment, etc.

To remove the burned fuel section from the maintenance pit a team of six men was used. By means of the overhead crane, a skip loaded with wet sand was lowered into the maintenance pit adjacent to the sand-covered rod section. The six men, using 24 foot long tools, each working for one minute only, raked the fuel section onto the skip and covered it with sand. The skip was then lifted by the overhead crane and placed on a previously prepared and partially shielded float for removal to the disposal area.

FIGURE 5. A disposable plastic suit, a plastic hood and an army respirator are shown. Under the suit is a cotton uniform.

The remaining sand in the maintenance pit contained fragments of the rod and radiation from this area was still intense. Forty men, two at a time, working for 1½ minutes each used long-handled tools to shovel the sand into prepared garbage cans. Radiation at contact with these cans was 200 R/hr.

Most of the residual contamination in the maintenance pit and on top of the reactor deck plate was removed by a special vacuum cleaner and by lifting the paper and plastic lining which had been placed over these areas before the accident.

Offices, auxiliary rooms and the basement areas were sealed from the main reactor hall by altering the ventilation system and air locks. These areas were then decontaminated by teams of workers employing large quantities of detergents, hot water, mops and wipers. All surfaces were washed two or three times in order to reduce the contamination to acceptable levels. Floors were given additional treatment.

When these areas could again be used normally, efforts were directed towards cleaning the main reactor hall. A civilian building maintenance contractor was hired to decontaminate the high sections of the building. The men, who were steeple jacks, had no previous experience in radioactive work and were therefore given a short course of instruction on radiation hazards control before beginning work. During their employment the men were tested daily for inhalation of radioactive materials. Urinalysis indicated that the quantities of fission products inhaled were negligible.

Decontamination of the reactor hall was continued by all available personnel. The methods used were quite simple. Walls, ceilings and equipment were washed several times by hand with a solution of a phosphate-base detergent in hot water. Electronic equipment was decontaminated by a vacuum cleaner and by wiping carefully with a damp but not wet cloth. Wet cleaning methods could not be applied to certain delicate and special instruments and, after vacuum cleaning, the residual contamination was accepted if it was not excessive. Equipment showing more than 5000 cpm after decontamination was sent to the central decontamination centre for more thorough treatment.

Except on the first night full plastic dress, rubber gloves beneath cotton gloves and respirators were worn for all operations in the reactor hall. (Figure 5.) Personal radiation exposures were measured by film packs and pocket do-

simeters. Individuals entering the reactor hall wore Canadian Army anti-gas respirators, and when entering other areas, wore a modified Comfo dust mask. (Figure 6.)

The army respirator gives complete protection against all radioactive particulate air contamination and is effective against many gases. The filter canister contains a pleated paper absolute filter backed by activated charcoal. The modified Comfo dust mask is much more comfortable to wear and consequently is more popular. The filter is made from pleated filter paper and the protection afforded against dusts and smokes is roughly the same as that of the army type. Because it contains no charcoal it is effective only against particulate air contamination and it is strongly suspected that radio-iodine will under certain conditions, penetrate the filter.

Cases of internal contamination of personnel occurred (Table I) and numerous complaints were made about the effectiveness of the respirators. A careful study showed that many individuals who had received internal contamination had not worn their masks properly. Some under the stress of the moment lifted the respirators from their faces in order to speak to fellow workers. Others held them away from their faces to relieve respiratory distress after rapidly climbing stairs.

The incidence of external body contamination, even during the emergency period, was never serious. The plastic suits worn over normal white clothing gave very good protection if workers were properly dressed and undressed. Specially trained men assisted in all dressing and undressing when plastic suits were used. In the early stages of the work, it was not unusual to find up to 200 mR/hr of loose contamination on the outside of these suits.

One of the major problems associated with the decontamination was the proper control of supplies, equipment and instruments. Quantities of salvageable material were unintentionally placed in waste disposal bags by inexperienced labour crews and subsequently lost. The need for a well organized and well trained labour force to do this work is important. The use of temporary workers not properly trained and instructed in the handling of such material can in a very short time cause the loss of thousands of dollars of recoverable items. Planning for emergencies and training of selected personnel will result in major economies.

In severe reactor accidents it is quite possible that change rooms may become unusable, either from contamination or from radiation. Both the NRU accident and the NRX accident in 1952 had this result. Within minutes of the accident the change rooms were unusable and remained so for several weeks. Without well controlled change areas, contamination control, as well as personnel and equipment control, becomes almost impossible. A trailer or portable type of change room which could be located near the most convenient exit from the reactor hall would have been invaluable. A mobile unit of this type has been designed at Chalk River and is now under construction.

FIGURE 6. Left: Canadian army anti-gas respirator. Right: Modified comfo dust mask.

TABLE I

Bioassay Results May 24–July 4, 1956

Total tests	1362
Individuals tested for new fission products except radio-iodine and cesium showing:	
greater than 150 dpm/urine output for 24 hours	43
greater than 1500 dpm/urine output for 24 hours	8
greater than 15000 dpm/urine output for 24 hours	0
Individuals tested for radio-iodine showing:	
greater than 2580 dpm/urine output for 24 hours	102
greater than 25800 dpm/urine output for 24 hours	33
greater than 258000 dpm/urine output for 24 hours	10

Note: Less than 1 per cent of new fission products was Sr^{90}.

Other items of equipment that would be most useful during accidents of this kind are:

(1) Portable remote-controlled radiation monitors.
(2) Easily portable shielding walls which can be installed with negligible radiation-exposure time.
(3) Portable ventilation systems complete with absolute filters.
(4) Efficient machines for decontamination of walls, ceilings, and floors.
(5) Well-shielded but easily-handled disposal containers.

Several of these devices are being developed at Chalk River.

Planning for a Radiation Accident*

EUGENE L. SAENGER, M.D.

College of Medicine, University of Cincinnati, Cincinnati, Ohio

THE PURPOSE of this paper is to describe rational approaches to potential radiation accidents so that in the event of emergency a well organized plan can be immediately put into operation. Radiation accidents do not only involve the one or several individuals directly exposed. Hazards may be encountered by others in the immediate vicinity and by the population either in the vicinity of an accident or even at some distance away. Serious psychological problems of public panic may occur; the entire situation is often one of confusion.

A radiation accident may be defined as an unforeseen occurrence, either actual or suspected involving exposure of or contamination on or within humans and the environment by ionizing radiation. The accident will be considered as occurring within a short time period of seconds up to several days. Chronic occupational or other long term exposure will not be considered.

The probability of the occurrence of such accidents is small since in eleven years in the AEC program employing over 300,000 people, there have been only 77 accidents or incidents directly involving radiation.[1] Two incidents occurred in non-AEC installations but are described by Hayes.[2] Some of these involved accidents to equipment, material or premises but did not involve people. There have been only four fatalities due to radiation from 1945 through 1958 in this group. There have been only 35 additional individuals who have lost time because of radiation injuries. The occurrence of accidents from 1945 through 1958 is shown in Table I. Until 1957–58 the number of accidents appeared to be rising, however this observation is probably due to the expansion of the entire AEC program.

There are two ways in which humans can be involved by radiation. The source of radiation may be outside of the body so that the radiation strikes the individual and is absorbed depending upon its physical characteristics. Radiation from x-ray generators, particle accelerators, sealed sources of radioisotopes and reactors are examples of this type. The radiation may be beta, gamma, or neutron. In particle accelerators other particles such as deuterons, alphas, etc., can be found. The Los Alamos,[3] Argonne,[4] Oak Ridge,[5] Russian,[6] and Yugoslavian[7] reactor accidents were of this type, i.e. exposure only to external radiation.

The second way in which humans can be involved is by contamination with radioactive nuclides. These radionuclides can be deposited on the skin, inhaled, ingested or enter the body through wounds or through the intact skin. Radioactive nuclides may also be formed within the body following exposure to an external source of neutrons. Those most commonly detected are Na^{24} and P^{32}. Individuals may be involved both from external radiation and by radioisotope contamination.

In order to prepare a suitable plan for dealing with such an accident, one should list all of the sources of radiation which can be found in his installation. The type of accident which can be anticipated follows readily. (See Table II.)

One must then estimate other factors which may bear on the anticipated accident (See Table III). For example at a reactor site, external radiation, neutron exposure, and contamination of people and the environment should be anticipated. As mentioned above most reactor accidents to date have been problems only in external radiation. However the accident at Windscale No. 1 Pile[8] resulted only in contamination of the English countryside with radioiodine-131. There was no personnel damage. The significant problem was to restrict milk distribution to the populace because of the high levels of I^{131} therein. The cycle involved escape of I^{131} fumes through the stack, transport by air, settling onto plant life, ingestion of plants by cows and finally production of I^{131} milk.

In an uranium ore processing plant or fuel reprocessing plant, one must be alert for inhalation exposure, skin contamination, incorporation of radionuclides into the body and widespread environmental contamination. External exposure to radiation will usually be minimal.

Depending on the particular installation, all possible combinations and permutations of the

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This work from the Department of Radiology and Radioisotope Laboratory was supported under contract AT(30-1)-2106 with the Division of Biology and Medicine, U. S. Atomic Energy Commission.

above hazards may be encountered. So long as the particular situation is carefully analyzed initially and reviewed periodically, an adequate emergency program can be in constant readiness.

One of the most difficult decisions in the accident is the decision as to whether the accident truly has taken place. In large installations where there are multiple area monitors, this problem is not so important. In smaller installations where radiation hazards are generally less, the problem is more serious. For example the extent of the iridium-192 incident in Houston[1] was not clearly recognized for several days. In this situation, although there was no significant radiation damage to people, the environmental contamination, cleanup, publicity and psychic trauma resulted in large financial losses. Thus it is essential, particularly where radioisotopes are involved, to sound the alarm immediately. If there is no evidence of radioactivity after an emergency survey, little is lost. On the contrary, once radioactivity is widespread, all problems connected with the incident seem to increase exponentially.

A proper emergency program consists of several important, closely interrelated aspects, all of which must be handled simultaneously. There are essentially three groups who have specific responsibilities. These are management, medical and industrial hygiene (including health physics). The management group once notified is concerned with general plant supervision, notification of authorities such as AEC, Public Health, etc., and most important publicity. The medical group is responsible for prompt handling of all injured and potentially injured people. The health physics group is responsible for determining contamination, estimating doses, evaluating what really happened and advising management and medical groups concerning same. The mutual cooperation of these groups is of the utmost importance .

In smaller installations there may not be a fulltime physician. It is important that physicians acquainted with nuclear medical problems be available both for emergency care and for long term care of injured people. Also the duties of health physicist may be only one of the responsibilities of the industrial hygienist. The responsibilities and functions of both physicians and industrial hygienists must be carefully coordinated and frequently reviewed. The plant nurse should be trained to aid in this type of emergency.

The immediate accident plan within the installation should be as follows:

1. Evacuate exposed personnel from accident area and provide first aid.

TABLE I

AEC Radiation Accidents[1]

(1945–1958)

Criticality incidents	11
Reactor incidents	9
Contamination	21
Fires	12
Explosions	4
Transportation	6
External personnel exposure	14
Total	77

TABLE II

Common Sources of Radiation and Possible Accidents Which Can Occur

Source	Type of accident which can occur		
	External radiation	Radio-isotope	Mixed
Reactor	gamma, neutron	yes	yes
Fuel element mfg	minimal	yes	yes
Fuel element reprocessing	gamma	yes	yes
Radioisotopes mfg. processing use	yes	yes	yes
Sealed sources (not leaking)	yes	no	no
Sealed sources (leaking)	yes	yes	yes
Particle accelerators	yes	yes	yes
X-ray generators	yes	no	no

TABLE III

Evaluation for Preplanning for Radiation Accidents

1. Types of radiation sources
2. Levels of radiation
   a. Energy
   b. Curiage
   c. Dose
3. Routes of dissemination
   a. External source
   b. Radioisotope—Air, Water, Buildings
4. Groups of people involved
   a. Those directly involved
   b. Other workers
   c. Police, Firemen, other helpers
   d. General public

2. Notify management, medicine and industrial hygiene.
3. Close off radiation area.
4. Confine and survey all possibly contaminated people.
5. Evaluate situation:
   a. Contamination,
   b. Neutron exposure, and
   c. Level of radiation exposure.

The evacuation of patients and first aid for thermal burns and other trauma is merely the

TABLE IV

Emergency Supplies for Contamination Accident

For Patients
Large paper bags for clothes
Large sheets of plastic or cloth
Cotton blankets or gowns
Paper foot covers (grocery bags)
Labels and tags
Marking crayons
Laundry bags
For Decontaminators
Coveralls, surgical scrub suits and gowns
Caps
Shoe covers—latex or plastic overshoes
Rubber gloves—industrial, surgical, obstetric
Dust respirators

exercise of common sense. One word about evacuation is important. If workers think something is wrong in a radiation area, speedy evacuation may be the difference between life and death. Each potential hazard should be discussed with them in this respect. There is little doubt that fast footwork was responsible for the survival of employee "A" in the Oak Ridge incident.[5] In a large installation notification of management and health physics groups will be done by automatic radiation alarm systems. In smaller installations, anyone who recognizes the problem can and must carry out this notification.

The radiation area should be closed off at once. If there is possible radioisotope contamination, air conditioning and ventilating systems must be closed and sealed. In sites of possible radioisotope contamination, all potentially exposed individuals who are ambulatory should be taken to appropriate areas for surveys. A list of supplies to facilitate this survey are shown in Table IV. With these supplies, people who are contaminated can strip while standing on the sheets. They may then cover themselves with blankets or gowns and go to a decontamination area. The sheets with contaminated clothing can be folded inwards and placed in labeled bags.

The evaluation of the emergency situation is often difficult. The physician and health physicist must answer three questions: (1) Is there radioactive contamination? (2) Has there been exposure to neutrons? (3) What level of radiation exposure have injured people received? The existance of contamination can be quickly checked by survey meters. The presence of neutron exposure can be anticipated by the nature of the installation, by area monitors and by immediate blood analysis for Na^{24}. The estimation of radiation dose is a problem to which an accurate answer is not always possible but is of great importance for therapy of injured individuals. If the health physicist and consultants can reach a reliable estimate, even in order of magnitude, these values will be most helpful to the physician. Necessary outside consultants should be summoned.

The immediate accident plan just described is one of particular importance for the medical team. Parallel programs for management and health physics will be as follows:

MANAGEMENT

1. Mobilize auxiliary help for medical and health physics team.
2. Take necessary action in event of fire and explosion.
3. Advise AEC and local safety and public health authorities.
4. Prepare immediate press, radio and television bulletins, giving necessary precautions and reassurance as indicated.
5. Alert community hospital facilities as directed by physician.

HEALTH PHYSICS GROUP

1. Close off radiation area. If contamination is a factor, the area must be sealed.
2. If contamination is present shut off air conditioning and ventilators insofar as possible.
3. Begin immediate radiation survey with mapping.
4. Assist medical group in obtaining surveys of contaminated individuals.
5. Do area wipe tests.
6. Prepare equipment for collections of body fluids, wipes of individuals, etc.
7. Attempt reconstruction of accident.

In any accident the time problem is critical. Particularly when contamination is present, radioactivity can be disseminated very rapidly. For example, in a spill of 12.5 mg of radium at Children's Hospital in Cincinnati, the broken source was discovered at 10:30 one morning.[9] The technician replaced the broken source in its container. A contaminated sheet was carried into another room and placed in a linen hamper. The responsible physician was notified about 45 minutes later. Within two hours the department was widely contaminated even though the room in which the accident occurred was sealed off.

For planning purposes the time periods should be divided as follows:

1. Immediate emergency—0–12 hours,
2. Later emergency—12–72 hours,
3. Decontamination and repair—days-1 month,
4. Long term follow-up.

The emergency period may be defined as the time after the accident has occurred during which events are out of control. As confusion

becomes minimized and an orderly plan of procedure is put into operation, the emergency is terminated. This period can be shortened by previously prepared plans of action. The term is used here in contra-distinction to a clinical medical emergency which can occur at any time.

The Problem of Radiation Injury

Of all problems created by a radiation accident, the most important is that of the exposed individual. Having followed the patient to the dispensary after evacuation from the accident site, it is now necessary to plan for his further care within the first several hours.

The non-contaminated individual should be put to bed. All samples such as clothes, blood, urine, feces, vomitus, etc. should be saved and labeled with time and date. A routine hemogram should be obtained. If neutron exposure is suspected, 10 ml of heparinized blood should be taken. An aliquot should be counted for sodium-24 activity. A urine sample should be analyzed for Na^{24} also. If a dose of more than 100r is suspected the patient should be hospitalized. A brief history and physical examination should be carried out. Baseline hemogram values from the occupational medical file should be available. Necessary emergency surgery should be done.

The problem of the contaminated individual is similar but somewhat more complicated. He should be reasonably well decontaminated after first aid has been given.[10] Personnel aiding in decontamination should be provided with adequate protective clothing.

In an installation where contamination is likely to be a problem, a decontamination room in the dispensary is helpful. This room should be equipped with an autopsy table so that radioactivity can be confined during the cleanup. Stall showers should be available for ambulatory individuals. If contaminated wounds are present surgical excision is indicated as soon as possible. The health physicist is an integral part of this phase since he must guide the surgeon in the extent of excision of wounds.[10]

The need for hospitalization of exposed individuals may be difficult to determine. Lacking accurate dose estimates, many individuals may seem to need hospitalization and may even demand such care. In general one need not hospitalize anyone whose total dose is less than 50 rem. Patients whose dose is of the order of 100 rem may be managed well without hospitalization if facilities are limited. This group of patients requires careful follow-up however, both for the protection of the individual and in order to provide data for compensation if justified.

The hospitalized patient should be given sedatives and antiemetics as indicated. If vomiting and diarrhea are marked, parenteral fluids should be administered. A complete history and physical examination should be done. Daily blood counts are required. All urines, feces, etc., must be saved. The patient must be guarded to prevent his losing large amounts of blood to physicians for "studies". In questioning many physicians skilled in nuclear medicine and health physicists, it is difficult to contemplate the need for over 20–30 cc of blood per day for useful study purposes.

If at all possible the hospitalized patients should be bedded in separate wards or rooms. If a group of potentially injured patients remain together, their symptoms tend to follow a herd pattern and there is an increase in apprehensiveness and attendant symptoms of weakness, nausea, vomiting, etc. The initial symptoms of the acute radiation syndrome are nausea and vomiting followed by a latent period of 1–18 days depending on the dose received. Since the above symptoms may occur with fright, separate confinement of the patients is essential especially since change in symptoms may be an important aid in estimation of dose to the patient.

The diagnosis and indicated treatment of the patient exposed to total body radiation depends on a clear understanding of the acute radiation syndrome in humans. The knowledge of this syndrome has been clearly described by Gerstner.[11] The syndrome may be sub-divided into three types: the cerebral type, gastrointestinal type and hematopoietic type. The estimated dose ranges to produce these manifestations are shown in Table V.

The recent death of a patient at Los Alamos is a good example of the effect of massive irradiation. A chemical operator received a dose of 12000 rem ± 50 per cent of neutrons and gamma rays at 4:30 pm.[12] He ran out of the operating

TABLE V

The Acute Radiation Syndrome

	Cerebral form	Gastrointestinal form	Hematopoietic form
Threshold dose	2,000 r	500 r	100 r
Characteristic signs and symptoms	Convulsions; tremor; ataxia; lethargy	Diarrhea; fever; disturbance of electrolyte balance	Leukopenia; purpura; hemorrhage; infection
Time of death	Within 2 days	Within 2 weeks	Within 2 months

area calling that he was burning up. Within one minute he showed mental confusion and ataxia. At ten minutes there was nausea; profound shock, nausea and diarrhea occurred in 30–45 minutes. Vigorous supportive therapy prevented immediate death. After 30 hours there was a rapid downhill course with death occurring about 35 hours after exposure.[13] At present we are not able to offer life saving therapy for such a patient.

The gastrointestinal form of death is illustrated by the clinical course of a scientist exposed during a fission experiment.[3] He received an estimated dose of about 2000r of combined neutron, gamma and x-radiation. He had nausea within a few minutes and vomited before reaching the hospital 60 minutes later. Vomiting increased within the next few hours and there was one loose stool. From 12 hours to the sixth day the patient improved. At that time rapid deterioration occurred with fever, nausea, vomiting and diarrhea. The white blood cell count fell precipitously. There was severe paralytic ileus and abdominal distention. In spite of antibiotics, blood transfusions, parenteral fluids and gastric suction, there was rapid downhill course with circulatory collapse on the eighth day and death on the ninth day. At autopsy the mucosa and submucosa of the entire small bowel was destroyed.

The hematopoietic form occurs with exposures below 500 rem.[11] After prodromal symptoms of weakness, headache, nausea and vomiting for 1–3 days, there is a latent period of 2–3 weeks. From 18–24 days there is a severe bone marrow depression shown clinically by general debility, insomnia, fever and bleeding. Recovery begins with 5–7 weeks.

The very brief summaries above are those of patients who have had much supportive therapy and excellent hospital care. One may assume that in patients who have received less than 500 rem, with excellent care recovery will occur in most patients. In patients who receive 500–1000r such a favorable result cannot be anticipated unless bone marrow transfusion is considered. The results of such therapy as used by G. Mathe and his colleagues were described recently.[14] One patient who received in excess of 1000r died. Four others recovered and it is thought that the marrow transfusions were in large part responsible for their recovery. The technique of bone marrow transfusion is especially difficult under the most favorable circumstances. When several individuals require such therapy, the technical problems become even more difficult. In addition there are certain hazards of the therapy itself and one should not use it unless the indications are definite. It is therefore essential that every effort be made to provide the physicians with as accurate an estimate of dose as can be made. Here the role of the industrial hygienist and health physicist is of the greatest importance.

The problem of the contaminated patient also requires close cooperation between health physics, the plant physician and the hospital. If the problem is only one of contamination, some decontamination can be carried out at the installation. If however, there are complicating injuries such as fractures, thermal burns and wounds, it is quite likely that one or more individuals who still have significant contamination will arrive at a community hospital for care. The hospital personnel will not understand how to deal with contamination and cleanup unless suitable emergency plans have been made prior to patient admission. The most suitable place for decontamination of a non-ambulatory patient is the stainless steel autopsy table. Larger nuclear facilities have such tables in their emergency treatment areas.[10] It may be necessary to bring patients through the autopsy room of the hospital in order to effect decontamination. If such a procedure is not possible some type of emergency area must be improvised. If the admitting room, operating rooms and hospital personnel are contaminated, the areas and personnel cannot serve other patients.

In radiation accidents, there are two situations in which information about radioactivity of the patient is important. One is following exposure to neutrons. In this situation, depending on the neutron spectrum and dose, various elements in the body will be made radioactive. The principal gamma emitting radioactivity will be Na^{24} and P^{32}. Exposure to alpha, beta and gamma radiation does not produce this type of radioactivity. The second is following ingestion, inhalation or penetration of radioisotopes.

If any of these nuclides are gamma emitters, their effective and biological decay can be followed by placing the patient in a device known as a total body counter. These counters are of two types. In one the patient is surrounded by a liquid scintillator or multiple crystals with multiple photoelectric cells connected to a readout device.[15] Such a counter gives an excellent indication of total activity but does not permit accurate indentification if several gamma emitters are present. The second consists of an iron shielded room to cut down on background in which is a large scintillation crystal and phototube connected to a multichannel pulse height analyzer.[16] This device permits determination of total activity and also determination of the several gamma emitters by pulse height analysis.

Access to such a counter facilitates diagnosis and treatment particularly if there is incorporation of gamma emitters within the body. These counters are now in construction or operation at certain large AEC installations. Many authorities believe that these counters should be available near hospital facilities wherever these accidents are anticipated.

Finally serious thought should be given to the problem of publicity. In several accidents within the past few years, there was a lack of free communication between the press, radio and television with the installation where the accident occurred. The result of the impasse was a rash of speculation and rumor which at times almost resulted in public and inplant panic. As a result of the Sylvania thorium fire in July 1956 and the Y^{12} accident at Oak Ridge in June 1958, certain techniques were evolved which were quite successful. An individual experienced in public relations was put in charge of all releases. Factual statements were provided to all inquiries giving brief accurate information promptly. The approach is similar to that of the airlines following a crash. If the pertinent facts are made public promptly, within the ensuing 2–3 days the excitement dies down. If however, little or nothing is released then rumors start. These may include speculations that there has been a nuclear bomb, that all the drinking water is radioactive, etc. The implications are obvious. The physicians and health physicists must provide regular bulletins to the public information officer but should not give direct interviews. If bulletins are supplied more rapidly than requests for information, the press will be cooperative; if not they will dig for their own scoops and feature stories and the entire affair will be greatly prolonged.

In summary one must anticipate by careful pre-accident planning for all possible factors in the particular installation. With careful organization and training of both plant and community health facilities, the confusion of radiation accidents can be significantly decreased. The role of the industrial hygienist is a most important one both in planning and in the event of an actual accident.

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Progress of the Automobile Industry in Regard to Automobile Exhaust and Air Pollution*

JAMES M. CHANDLER

Vehicle Combustion Products Committee, Automobile Manufacturers Association, Detroit, Michigan

THE WORKING theory in Los Angeles in regard to smog formation[1] is that hydrocarbons and oxides of nitrogen react in sunlight to form ozone and smog irritants. Automobile exhaust gases contain both unburned hydrocarbons and oxides of nitrogen.

In recognition of the "working theory" and the increasing number of attacks of photochemical smog in Los Angeles the Automobile Manufacturers Association formed the Vehicle Combustion Products Committee in December of 1953. This committee was assigned the task of determining the role of automobile exhaust in air pollution and was also instructed to devise means for eliminating those automobile exhaust emissions that were deemed undesirable in regard to air pollution.

From the initial analysis of the problem it was decided that several areas of investigation should be pursued vigorously and concurrently, contrary to normal research programs which follow an orderly succession of defining the problem, developing instrumentation, designing and building test prototypes, evaluating test prototypes, and repeating this cycle until the final solution is achieved. A variety of considerations led to this decision:

1. It would be necessary to develop instrumentation for analyzing automobile exhaust gas in terms of ppm and pphm. Available instrumentation consisted primarily of the Orsat Analyzer which measured CO, CO_2, oxygen and nitrogen only as low as 0.2% by volume. The instrumentation needed to detect quantitatively the hydrocarbons in automobile exhaust gas did not exist, and would have to be invented and developed if any useful work were to be done toward eliminating hydrocarbons from automobile exhaust.
2. A study would have to be made to determine how people operate automobiles in terms of the percentage of time spent when the car is idling, accelerating, decelerating and cruising, and values for the average cruising speed and rates of acceleration and deceleration would have to be determined for a large representative population of cars so that the performance of an average car could be determined. For such a study it was obvious again that new instrumentation would have to be designed and built.
3. Presupposing success in the development of instrumentation and analytical techniques for determining hydrocarbons and oxides of nitrogen during various engine operating cycles, it would then be necessary to conduct a full scale exhaust gas field survey where a large representative population of cars could be tested in a uniform manner and an exhaust gas analysis obtained for each car.
4. By careful analysis of the traffic survey and field survey data it might then be possible to define exhaust gas emission in terms of concentrations of components in the exhaust and the rate of emission so that the total emission of pollutants to the atmosphere could be determined for any representative car population.
5. It was also deemed necessary to study the composition of exhaust gases from new cars adjusted to factory specifications and also of used cars which are out of adjustment due to lack of maintenance to determine whether or not a real exhaust emission difference exists, and if so, what steps could be taken by the manufacturer to reduce this difference and what steps could be taken by the car owner to keep his car maintained to a minimum level of hydrocarbon and oxides of nitrogen emission.
6. It would be necessary to conduct a thorough literature search for existing knowledge which might be of help.

Implementation of Industry Program

It was immediately recognized that in the area of determining exhaust gas composition the Coordinating Research Council (CRC) was avail-

* Presented April 30, 1959 at the Air Pollution Session of the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois.

able to the industry since both the automotive and petroleum industries who support the CRC were interested in determining exhaust gas composition. A Group on the Composition of Exhaust Gases was thus formed within the CRC and four panels were established:

1. Sampling and Analysis Panel—To develop the necessary instrumentation and analytical techniques for determining exhaust gas composition.
2. Variables Panel—To study the effects of fuel, lubricant and engine variables on exhaust gas composition.
3. Field Survey Panel—To conduct test programs in the field to determine the exhaust gas composition of representative car populations.
4. Literature Survey Panel—To survey all existing literature and distribute the resulting bibliography to the membership of the Group on the Composition of Exhaust Gases.

Within the Automobile Manufacturers Association a Vehicle Combustion Products Committee was formed in December of 1953 under the auspices of the Engineering Advisory Committee and was organized as follows:

1. Induction System Panel—To develop induction system devices to prevent unburned hydrocarbons and oxides of nitrogen from entering the engine exhaust system.
2. Exhaust System Panel—To develop devices in the exhaust system for removing unburned hydrocarbons and oxides of nitrogen in the exhaust.
3. Traffic Survey Panel—To develop necessary instrumentation and conduct a full scale traffic survey in a representative car population to determine how people drive.
4. Data Analysis Panel—To analyze all pertinent data as they become available and to keep the parent committee up to date as new data are developed.
5. Carbon Monoxide Panel—To develop instruments and procedures for measuring carbon monoxide concentration in passenger compartments to help in the design and location of vehicle exhaust systems.
6. Visible Smoke Emission Panel—To aid in the development of enforcement techniques such as training films and brochures for identifying automobiles with excessive visible smoke emission.
7. Exhaust System Engineering Panel—To study the effects of exhaust system engineering on carbon monoxide concentration in the passenger compartment.
8. Medical Panel—To study health aspects of exposures to automobile exhaust.

Results

The Sampling and Analysis Panel of the CRC, whose members come from both automotive and petroleum laboratories, has released several reports[2, 3] dealing with both grab and continuous techniques for analyzing hydrocarbons, oxides of nitrogen, carbon monoxide, carbon dioxide, etc. Work is continuing in the Sampling and Analysis Panel to refine the techniques for determining total hydrocarbons with the infrared analyzer. Work is also continuing under contract at the Bureau of Mines in Bartlesville, Oklahoma, with funds supplied by the automobile and petroleum industries, to develop gas chromatographic techniques[4] to identify specific hydrocarbons in automobile exhaust gas.

The Variables Panel of the CRC has completed a study of engine variables and their effects on exhaust gas composition and has released a report[5] dealing with this subject.

The Field Survey Panel of the CRC has conducted two field surveys in which over 350 cars were tested for exhaust emissions, and has recently released a report[6] with those results.

The Induction System Panel of the AMA accepted as work was initiated in this field several years ago, the then available data which indicated that over 60% of the total hydrocarbons emitted to the atmosphere from automobile exhaust came from the engine during deceleration when there is essentially no combustion. Without waiting as would be normal for the development of instrumentation and test techniques and the running of sufficient tests to determine similar information, the Induction System Panel initiated a vigorous cooperative program to develop a device to decrease hydrocarbon emission during deceleration. After several years of work two types of devices were perfected[7] and submitted in 1957 to the Los Angeles county Air Pollution Control District for evaluation.

By that time, however, the work of the CRC Panels and other automotive industry groups working on the problem indicated that only 20 to 30% of the total hydrocarbons emitted to the atmosphere from automobile exhaust came from the deceleration cycle rather than the 60% figure which had been assumed when the device development program was initiated. The application of a deceleration device thus appeared less promising in terms of reducing total hydrocarbon emission to the atmosphere from automobile exhaust.

The Exhaust System Panel has conducted some basic research into several types of afterburners for oxidizing hydrocarbons in the exhaust system.

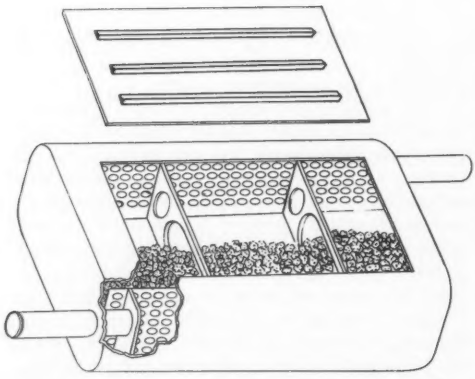

FIGURE 1. MX-2 Catalytic Converter. An example of the low temperature type catalytic converter.

FIGURE 2. A catalytic afterburner has been installed in place of the usual muffler. Note insulation to maintain temperature until combustion is accomplished. Unit is slightly larger than usual muffler.

In January of 1959 four technical papers[8, 9, 10, 11] on exhaust system devices were presented to the Society of Automotive Engineers at the annual meeting in Detroit. These papers described three types of exhaust system devices, the low temperature catalytic converter, the high temperature catalytic converter, and the non-catalytic (direct flame) afterburner. The following discussion of these three exhaust system devices is based on the SAE papers mentioned above.

An example of a low temperature catalytic converter is shown in Figure 1. In this system exhaust gases already containing added air enter the converter through the lower left hand inlet pipe, flow crosswise through the catalyst bed where the hydrocarbons are oxidized to carbon dioxide, and then exit through a standard tailpipe attached to the outlet at the upper right corner. A small air compressor is installed on the engine to supply the additional air needed for combustion in the catalytic convertor. Figure 2 shows the catalytic converter installed replacing the standard production muffler on a 1959 passenger car. Figure 3 shows stabilized road load efficiencies obtained with several different catalysts tested in the low temperature catalytic converter system. It can be seen that catalyst D represents substantial improvement over catalysts A, B and C which were tested in the earlier stages of the development program. It should be emphasized that these efficiencies were obtained after stabilized temperatures were achieved during constant road load operation at the various speeds indicated. In practice the converter actually operates at somewhat higher efficiencies because of the frequent normal driving accelerations and decelerations which tend to increase the temperature of the catalyst bed.

As part of the development program on the low temperature catalytic converter some tests were run at the Stanford Research Institute "Smog Chamber" under the auspices of the Air Pollution Foundation. In this facility the exhaust

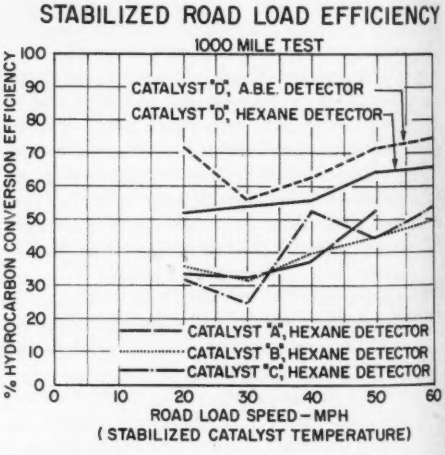

FIGURE 3. Chart of stabilized road load efficiencies of four low temperature catalysts.

gas from an automobile on a chassis dynamometer is blended with pure air, then fed to an irradiation chamber and subsequently to a panel of students who determine the eye irritation characteristics of the mixture in the smog chamber. Figure 4 shows the results of the testing with the catalytic converter. It should be emphasized that a standard tuned car did not produce any significant eye irritation until the car was detuned by richening the fuel flow and using more deceleration exhaust to charge the smog chamber. Under these test conditions the severe eye irritation obtained without the catalytic converter was effectively eliminated when the converter was installed on the test car.

Several major problems remain to be solved before the low temperature catalytic converter system can be classed as commercially acceptable. The warm-up of the converter on cold starts needs substantial improvement. In addition it would be desirable to have higher over-all operating efficiencies and longer catalyst life. At the present stage of development, it is estimated that the catalyst will show over-all efficiencies between 60 and 80% for 10,000 to 12,000 miles of driving.

An example of a high temperature catalytic converter is shown in Figure 5. In this design the exhaust gases flowing into the converter cause extra air to flow through a venturi and orifice arrangement. The exhaust gases and extra air then pass down through a catalyst bed and out to the atmosphere. Figure 6 shows the efficiency of this converter at 1,000 miles of operation for various driving conditions. Figure 7 shows estimated hydrocarbon reductions under a standard driving cycle for the high temperature catalytic converter. From these tests it was concluded that the life of the catalyst was between 10,000 and 12,000 miles, at a hydrocarbon reduction efficiency of approximately 75%.

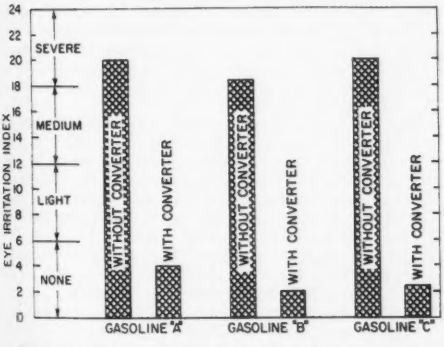

FIGURE 4. Comparison of index of eye irritation from irradiated exhaust gases from autos with and without use of catalytic converter. Tests of three types of gasoline under conditions of rich air/fuel ratio and disproportionate deceleration. Tests made using Stanford Research Institute Smog Chamber.

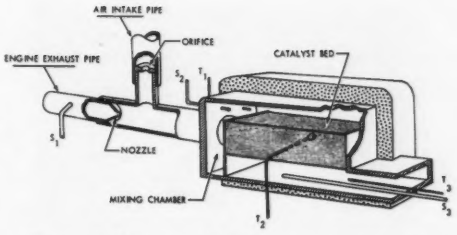

FIGURE 5. Diagram of high temperature converter system.

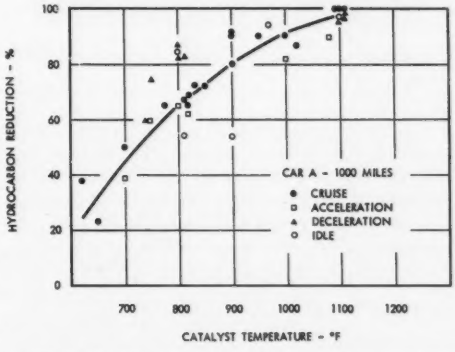

FIGURE 6. Efficiency of single high temperature converter at 1000 miles.

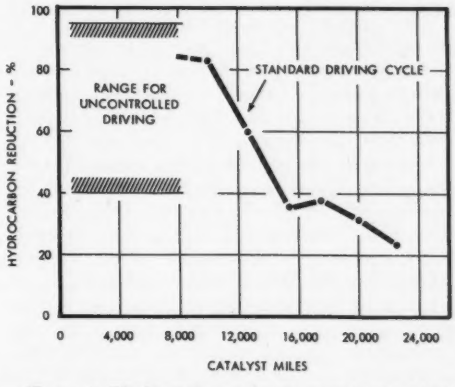
CAR A - ROAD EFFICIENCY BASED ON TEMPERATURE MEASUREMENTS
HYDROCARBON REDUCTION - %
RANGE FOR UNCONTROLLED DRIVING
STANDARD DRIVING CYCLE
CATALYST MILES

FIGURE 7. Hydrocarbon reductions under standard driving cycle with use of a high temperature catalytic converter on the exhaust.

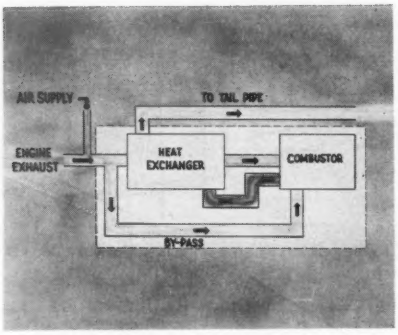

FIGURE 8. Diagram of a non-catalytic afterburner system.

Tests were also run with the high temperature catalytic converter in conjunction with a rich mixture carburetor in order to eliminate oxides of nitrogen as well as hydrocarbons in the exhaust system. It was concluded that this combination produced a 90% efficiency level in elimination of hydrocarbons, 75% reduction in carbon monoxide, and 90% reduction in oxides of nitrogen for 10,000 to 12,000 miles of operation. Major problems remaining to be solved with this type of catalytic converter include excessive exhaust system noise, bulkiness of the converter, and long warm-up time. It should also be noted that a car using the rich mixture which eliminated oxides of nitrogen suffered a 25% reduction in miles per gallon.

An example of a non-catalytic afterburner is shown schematically in Figure 8. In this design the hydrocarbons and carbon monoxide in the exhaust gases are burned in a combustion chamber with extra air. The high temperature gases from the combustion chamber then pass through a heat exchanger to increase the temperature of the exhaust gases entering the afterburner. Efficiencies of over 90% in removing hydrocarbons have been achieved with this device but again many problems remain to be solved. For example, it would be desirable to achieve cost reductions in the stainless steel design required for the heat exchanger and simplification of the control system required to prevent the unit from overheating. A reduction in over-all size is also considered essential before this unit could be applicable to present day passenger cars.

At the present time, the Vehicle Combustion Products Committee of the Automobile Manufacturers Association is evaluating samples of the low temperature catalytic converter system and will evaluate the high temperature catalytic converter and the non-catalytic afterburner as soon as prototype samples are made available. Several low temperature catalytic converter systems have recently been shipped to the Los Angeles county Air Pollution Control District, the Bay Area Air Pollution Control District and the California State Department of Health. Two of these converters are currently installed at the Los Angeles county Air Pollution Control District undergoing an evaluation test program which is being undertaken concurrent with the evaluation and development programs within the automobile industry.

The results of the current automobile industry program on the development of exhaust system devices will be reported as they become available.

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TETRAETHYL LEAD INCREASE

PUBLIC HEALTH SERVICE Publication No. 712, *Public Health Aspects of Increasing Tetraethyl Lead Content in Motor Fuel* is now available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. for 30 cents per copy. This publication is the report of a special committee appointed to consider this problem. While stating that full data were not available for a conclusive answer the committee made two conclusions and three recommendations. The report gives the supporting and background material.

The committee concluded: "(1) a change in the maximum concentration of tetraethyl lead in motor fuel from 3.0 to 4.0 cc per gallon would not increase the hazard involved in the manufacture and distribution of leaded gasoline, and (2) available data do not indicate that such a change would significantly increase the hazard to public health from air pollution."

The recommendations are, if the industry does increase the concentration of tetraethyl lead, that: "(1) The industry proceed cautiously with the increase so that the increase in any one year in the tetraethyl lead content of U.S. pool gasoline not exceed 0.25 cc per gallon, and that the concentration of tetraethyl lead in U.S. pool gasoline not be more than 3.2 cc per gallon at the end of five years. (2) The industry supply annually to the Public Health Service the domestic consumption figures for tetraethyl lead. (3) The Public Health Service, in collaboration with the manufacturers of tetraethyl lead, the petroleum industry, and other appropriate organizations, conduct studies to provide more definite data on levels and trends of atmospheric lead contamination in selected urban areas and of the body burden of lead of selected population groups. If these studies should bring to light new data of significance, the recommendations of the Committee should be reconsidered."

Epidemiological Findings Associated with Beryllium Extraction*

JAN LIEBEN, M.D., and FRANZ METZNER, M.D.

Division of Occupational Health, Pennsylvania Department of Health, Harrisburg, Pennsylvania

BERYLLIUM DISEASE is caused by the toxic effects of beryllium and its compounds. Over the past twelve years cases of beryllium disease have been reported from various industries throughout the United States. This disease manifests itself in acute or chronic form, or as a dermatitis. It is a disease of low incidence and affects only a small percentage of those exposed. To date, it is not known why certain persons are singled out by the disease.

The diagnosis is difficult and is frequently open to challenge. The lung findings can easily be confused with similar diseases such as sarcoidosis, "farmers lung", and other diseases caused by exposure to dust. The problem is further complicated by the fact that frequently the disease develops long after the exposure to beryllium or its compounds has ceased.

The epidemology and clinical findings of acute and chronic lung disease which originated in and around a beryllium extraction and fabricating plant in Ohio were first reported by VanOrdstrand,[1] DeNardi,[2] and Eisenbud and associates.[3] Several papers have been presented as a follow-up.[4, 5]

Several large groups of cases of beryllium disease have been reported from the fluorescent lamp industry.[6] Beryllium-containing compounds were used to coat fluorescent tubes and a small percentage of those exposed inside the plant as well as some persons who had lived in the neighborhood developed the disease.

Since 1952 Hardy and her collegues have collected information on more than 600 cases of all forms of beryllium disease, and have established a central beryllium registry at the Massachusetts General Hospital in Boston. A summary of the presently available information on beryllium disease and its control was recently presented.[7]

Around 1937 a beryllium extraction and metal fabricating plant started operations close to a rather densely populated area in Pennsylvania. Several industrial hygiene surveys were conducted, and the findings at the plant were reported in the literature in 1944,[8] and 1949,[9] and 1954.[10] Cases of beryllium disease resulting from in-plant and neighborhood exposure in and around this beryllium extraction and alloy fabricating plant in Pennsylvania were reported, first by Chamberlin in 1954[11] and by Hardy in 1955.[12] In October 1956, twenty cases of chronic pulmonary beryllium disease were reported from the above plant by Chamberlin, Jennings, and Lieben.[13]

In April 1957 a research grant to study the epidemiology of beryllium disease and the environmental conditions of the Pennsylvania beryllium plant was received from the National Institutes of Health. This study included an extensive twelve month air pollution survey of the area surrounding the beryllium plant. Simultaneously atmospheric concentrations of beryllium were studied in other areas of Pennsylvania where there were no beryllium plants, particularly in areas surrounding steel mills or soft coal burning installations. The air pollution survey results and problems encountered during the chemical and spectrographic analysis are being reported in separate papers.[14, 15]

This paper is a preliminary report of the medical and epidemiological findings of the survey and deals with the analysis of 58 cases of the chronic pulmonary form of beryllium disease, diagnosed over the past 12 years by local physicians and clinical experts from several medical schools of the eastern United States. It is assumed that the cases are related to exposure to beryllium either from employment or to living in the neighborhood of the plant.

The cases include three groups:

1. Those who had contact with beryllium as plant employees.
2. Those who handled contaminated work clothes of beryllium plant workers.
3. Persons who lived, or frequently visited, in the neighborhood of the plant.

Groups 2 and 3 may overlap to a certain extent.

The plant population fluctuated over the years and reached as high as 800 during the war years and went down to 250 at other times. Annual labor turnover amounted to nearly 100% during the initial years of plant operation, with

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association in Chicago, April 26, 1959.

a marked decrease during the past decade. It can be assumed that approximately 4,000 workers have been exposed in the plant over the last 20 years.

In 1958 a comprehensive search for the potential beryllium disease cases was undertaken. This search was started in the record room of the three local hospitals. All files containing information on established or suspected cases were studied. Those living, or relatives of those deceased, were visited in order to obtain exact information on their background, with emphasis on the occupational history. From those contacted, more names and addresses of persons with beryllium disease were obtained. Over 200 physicians in the neighborhood of the beryllium plant were contacted by letter in the course of case-finding efforts. Each hospital record was abstracted and the physicians who had treated the patients were visited.

Only cases which show the chronic or prolonged pulmonary form of beryllium disease are included in this paper. The acute cases, and their follow-up will be discussed in a separate paper in the near future.

Diagnostic Criteria for Inclusion of Cases

Roentgenographic evidence of beryllium disease on progressive films, physiologic impairment, histologic changes or laboratory findings on biopsy or postmortem lung specimens are a desirable prerequisite for the diagnosis of beryllium disease. Each of the tables lists separately several cases which are not completely documented because of absence of biopsy or autopsy studies. These cases, however, closely follow the general pattern of the other cases of beryllium disease.

To establish the diagnosis of beryllium disease or to exclude it in every instance is extremely difficult and upon reviewing cases several years later, considering new factors, additional data and progress of the disease revisions may have to be made.[17]

In-plant Beryllium Disease Cases

The beryllium plant processes the crude ore into beryllium metal, manufactures beryllium alloys, and machines some of these alloys. The fluoride process is used for the refining of beryllium.

Cases of beryllium disease occurred throughout the plant and include many occupations in the plant population. The earliest case we know of dates back to 1943, the last in-plant case developed in 1957.

This group includes 18 plant employees who developed the chronic form of beryllium disease, either while working at the plant or after cessation of exposure. Detailed findings are listed in Table I. The longest period between cessation of exposure to beryllium and its compounds in the plant and the onset of symptoms of beryllium disease is 15 years. Fourteen of this group

TABLE I

In-plant Cases of Beryllium Disease

No.	Age group	Sex	In-plant assignment	Duration of exposure prior to onset of symptoms	Onset of symptoms	Latent period	Lung biopsy or autopsy (Histology)	Beryllium analysis of lung tissue
1	30–40	M	production	3½ years	1953	no	+	+
2*	30–40	M	production	not known			+	+
3	30–40	M	maintenance	2 months	1950	8 years	+	+
4	30–40	M	production	1 year	1954	7 years	+	+
5	30–40	M	production	5 years	1953	no	+	+
6*	60–70	F	production	3½ months	1950	5 years	+	not done
7	40–50	F	nurse	1¼ years	1954	8 years	+	negative
8*	30–40	M	production	4 years	1946	no	+	not done
9	40–50	F	maintenance	1¼ years	1948	3 years	not done	not done
10	30–40	F	production	1¼ years	1945	3 months	not done	not done
11	20–30	M	production	4 years	1956	no	not done	not done
12	20–30	M	production	7 years	1955	no	not done	not done
13	30–40	M	maintenance	3 months	1951	6 years	not done	not done
14	30–40	M	production	4 years	1955	no	not done	not done
15*	40–50	M	maintenance	1 year	1950	1 year	not done	not done
16*	60–70	M	production	9 years	1952	no	not done	not done
17*	60–70	M	maintenance	1 year	1943	no	not done	not done
18	60–70	M	production	15 years	1957	no	not done	not done

* Indicates patient deceased.

All persons listed have radiographic evidence of chronic pulmonary disease.

TABLE II

Cases of Beryllium Disease from Contact with Contaminated Clothing

No.	Age group	Sex	Duration of contact	Onset of symptoms	Latent period	Lung biopsy or autopsy (Histology)	Beryllium analysis of lung tissue
1*	50–60	F	5 years	1944	no	+	+
2*	30–40	F	1 year	1947	5 years	+	+
3*	40–50	F	6 months	1948	5 years	+	+
4*	50–60	F	9 years	1946	1 year	+	+
5*	50–60	F	5 years	1948	4 years	+	+
6	30–40	F	11 years	1956	no	+	+
7	30–40	F	1 year	1957	14 years	+	+
8	50–60	F	7 years	1953	5 years	+	negative
9	20–30	M	2 years	1949	4 years	+	not done
10*	10–20	F	2 months	1947	no	+	not done
11*	30–40	F	9 years	1943	no	+	not done
12*	40–50	F	12 years	1946	no	+	not done
13*	50–60	F	7 years	1945	no	+	not done
14	50–60	F	3 years	1954	6 years	not done	not done
15	10–20	F	2 years	1954	9 years	not done	not done
16	40–50	F	7 years	1955	3 years	not done	not done
17	40–50	F	2 months	1949	1 year	not done	not done
18	50–60	F	13 years	1954	no	not done	not done
19*	50–60	F	6 years	1947	no	not done	not done

* Indicates patient deceased.

All persons listed have radiographic evidence of chronic pulmonary disease.

are male and four are female employees. All persons showed the typical symptoms and clinical findings of beryllium disease.

Handling Beryllium Contaminated Clothing

This group includes eighteen females and one male who were exposed to beryllium dust for contact with, or by washing beryllium contaminated working clothes of plant workers (Table II). Most persons of this group also have lived or now live in the neighborhood of the beryllium plant, and had, additional exposure to beryllium contaminated air for many years. It is impossible, to estimate how much environmental air pollution contributed to the development of beryllium disease in this group.

Eisenbud and his group[3] have shown that home laundering of work garments can account for the inhalation of up to seventeen micrograms of beryllium per day. We assume that conditions in this population are not much different from those reported by Eisenbud. It should be noted, however that exposure to beryllium contaminated work clothes by house contacts has been reduced since 1955, and made almost impossible since the middle of 1958 when the double locker system in the plant was completed. This prevents the removal of working clothes, underwear, socks, or shoes from the plant premises. Therefore, exposure to beryllium contaminated working clothes no longer exists, and the cases in the above category are the result of beryllium exposure up to the middle of 1958.

Exposures to beryllium dust from working clothes ranged from two months to thirteen years and was followed by the development of typical chronic beryllium disease, either during the exposure time or up to fourteen years after cessation of the exposure.

This group includes chronic beryllium disease in two members of the same family. A boy aged three and a girl aged fifteen were exposed for two successive years to beryllium contaminated clothes in their home, while their father worked at the beryllium plant from 1943–1945. The onset of symptoms, characteristic of beryllium disease, occurred in the boy four years after exposure and in the girl after nine years. The diagnosis in the boy was confirmed by lung biopsy two years ago.

Neighborhood Cases

This group, with twenty-one cases of chronic beryllium disease (Table III), is the largest in

TABLE III

Cases of Beryllium Disease among People Living in the Neighborhood of a Beryllium Plant

No.	Age group	Sex	Distance plant–home	Duration of exposure prior to onset of symptoms	Onset of symptoms	Lung biopsy or autopsy (Histology)	Beryllium analysis of lung tissue
1*	60–70	F	4.3 miles	15 years	1953	+	+
2*	50–60	F	4.2 miles	19 years	1956	+	+
3*	50–60	M	4.0 miles	16 years	1954	+	+
4*	30–40	F	4.5 miles	10 years	1947	+	+
5*	40–60	F	4.2 miles	16 years	1953	+	+
6*	10–20	F	1.7 miles	6 years	1953	+	+
7	20–30	M	1.8 miles	13 years	1952	+	+
8	30–40	F	4.4 miles	18 years	1955	+	not done
9*	30–40	M	4.7 miles	12 years	1954	+	not done
10*	60–70	F	4.3 miles	16 years	1955	+	not done
11	60–70	F	3.7 miles	18 years	1955	+	not done
12*	70–80	F	5.0 miles	10 years	1947	+	not done
13	30–40	F	4.3 miles	18 years	1955	not done	not done
14	40–50	M	5.0 miles	14 years	1952	not done	not done
15	20–30	F	1.4 miles	19 years	1956	not done	not done
16	40–50	F	2.2 miles	11 years	1948	not done	not done
17	40–50	F	4.3 miles	15 years	1952	not done	not done
18	60–70	M	0.7 miles	17 years	1954	not done	not done
19*	60–70	F	1.4 miles	14 years	1951	not done	not done
20	60–70	F	3.7 miles	16 years	1953	not done	not done
21	70–80	F	3.5 miles	11 years	1948	not done	not done

* Indicates patient deceased.

All persons listed above have radiographic evidence of chronic pulmonary disease.

this series and these individuals never had, to our knowledge, any direct contact with beryllium as industrial workers, or by cleaning contaminated clothing, or by frequent contact with persons working in the beryllium plant. They lived or live in the more or less densely populated neighborhood of the plant. The direct distance from the beryllium plant to the homes where affected persons live ranges from 0.6 to 5.3 miles. This is considerably farther than distances reported by Eisenbud et al.[3] from the Ohio beryllium extraction plant. For better illustration and comparison see Tables IV and V. (Figure 1.)

The Ohio plant is located near a lake in flat terrain, whereas the Pennsylvania plant is located in the northern part of a shallow valley which stretches approximately in north-south direction. It is bordered by a chain of low hills in the west, and a chain of rather steep hills up to 1,140 feet in elevation in the east. The prevailing wind direction is from the northwest. The greatest population density is found at a distance of 2.5 to 7.5 miles south of the beryllium plant. A 7.5 mile radius around the Pennsylvania plant includes a population of 100,000 with 80% living south of the plant. According to Eisenbud's paper the Ohio plant neighborhood area was populated by 500 persons living within a quarter of a mile around the plant.

In order to determine the atmospheric concentrations of beryllium in the area surrounding the plant, and possibly relate these concentrations to the distribution of the neighborhood cases, a continuous air sampling program was initiated early in 1958. The air sampling instruments used were 20 high volume air samplers, which were set up throughout the area in a pattern as shown. (See Figure 2.) These samplers pull approximately 4,000 cubic meters of air through a fine mesh fiber glass filter during a 48-hour period. This air sampling program has been in operation for twelve months. It can be stated, at this point that the mean concentrations in the air were on many occasions, higher than the recommended limit of 0.01 micrograms per cubic meter suggested by the A.E.C.

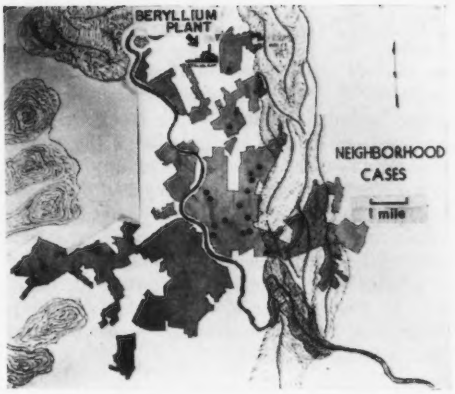

FIGURE 1. Location of neighborhood cases of beryllium disease with respect to site of beryllium plant.

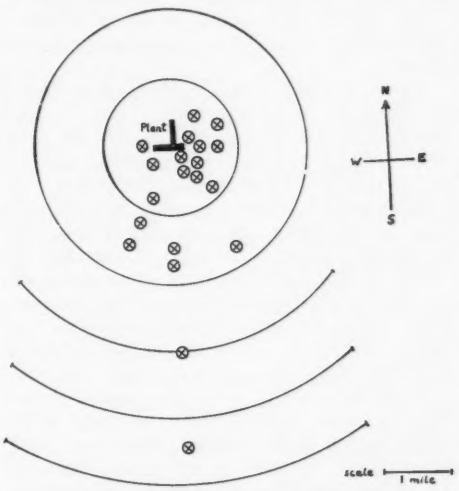

FIGURE 2. Location of air samplers.

TABLE IV

Neighborhood Cases in Pennsylvania

Distance from plant	No. of cases
0 to ½ mile	0
½ to 1 mile	1
1 to 1½ miles	2
1½ to 2 miles	2
2 to 3 miles	1
3 to 4 miles	3
4 to 5 miles	10
5 to 6 miles	2
Over 6 miles	0

TABLE V

Neighborhood Cases in Ohio*

Distance from plant	No. of cases
0 to ¼ mile	5
¼ to ½ mile	3
½ to ¾ mile	2
¾ to 1 mile	0
1 to 1½ miles	0
1½ to 2 miles	1
Over 2 miles	0

* From: *J. Ind. Hyg. & Toxicol. 31:* 282 (Sept. 1949)

In order to study non-occupational exposures further, post mortem lung specimens of residents from the area surrounding the plant were analyzed for beryllium contamination. This group included a physician, a truck driver, a housewife, in short a cross section of the general population. None had ever had any signs or symptoms of chest disease. They had died from other causes in a hospital a few miles from the plant, and twenty consecutive cases who were autopsied were studied. Each lung except that of a three year old child was positive for beryllium in much higher quantities than the lungs of persons who had lived in areas 70 miles distant from the plant.[11]

Discussion of Findings

An epidemiological study of beryllium disease in the neighborhood of a beryllium extraction and alloy fabricating plant produced findings, which are in many points similar to those reported from a beryllium extraction plant in Ohio.

So far, a total of fifty-eight cases of chronic beryllium disease have been found in and around the Pennsylvania plant and these include eighteen in-plant, nineteen contaminated clothing cases, and twenty-one neighborhood cases.

It should be pointed out that beryllium disease is not always easy to diagnose, but experienced physicians are able to establish the diagnosis and differentiate it from other diseases in most instances. The reported case series may contain a few cases where additional diagnostic procedures would be helpful, but it is felt that with the clinical findings, and with the careful evaluation of the epidemiological data, a rather high degree of accuracy in the diagnosis has been obtained. Post mortem examination in 19 cases out of 58 cases, which had the diagnosis of chronic beryllium disease established, or where beryllium disease was suspected, revealed valuable information and confirmation of the diagnosis.

The topographical structure of the area surrounding the plant, prevailing wind direction and distribution of the population may have contributed to the development of neighborhood cases in a rather limited area 3–5 miles southeast of the plant.

The air sampling program was not conducted during the actual time when air pollution is believed to have caused the neighborhood cases, but it can be conservatively assumed that similar or higher concentrations would have been found in the past.

It was recently pointed out[16] that safe non-occupational atmospheric beryllium concentrations in the neighborhood of beryllium-using plants can be attained by use of reasonably economic methods. Good industrial hygiene in the beryllium plant should minimize the danger of development of beryllium disease in the plant employees. Beryllium contaminated work clothes as a source of non-occupational exposure to beryllium dust, which led to the development of nineteen cases of chronic beryllium disease, no longer exists. All contaminated clothing is kept in the plant and cleaned in a special facility.

By observing these measures beryllium disease in and around beryllium plants can soon become a problem of the past.

Summary

An epidemiological study related to a beryllium extraction plant resulted in the finding of 58 cases of chronic pulmonary beryllium disease.

The cases fall into three groups: plant workers; relatives of plant workers who had to come in contact with berylluim contaminated clothing; and persons who live in the neighborhood of the plant.

The cases are described by group, sex, length of exposure, latent period and diagnostic criteria. The difficulties of the diagnosis of beryllium disease are discussed.

Mention is made of a simultaneously conducted air pollution study. The peculiar topographic and meterological conditions of the area which may have influenced the causation of the neighborhood cases are described.

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Chamber Monograph

PUBLIC HEALTH MONOGRAPH No. 57, *Exposure Chambers for Research in Animal Inhalation* by David A. Fraser, Ronald E. Bales, Morton Lippman, and Herbert E. Stokinger was published concurrently with the October 1959 issue of *Public Health Reports*. For readers wishing the data in full, copies of the monograph are on sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. The price is 40 cents per copy. When ordering specify Public Health Service Publication No. 662.

This monograph was prepared by the Occupational Health Program, U.S. Public Health Service. Drawing on the long experience of their own laboratories, other research centers and private investigators, the authors have presented chamber designs for various types of exposures and studies. Emphasis is placed on the fundamental requirements of the chambers and the reason for each design and special feature. Basic equations and formulas affecting performance and chamber dynamics are given. There are more than 80 references and 30 illustrations and tables.

This publication should be of considerable value to investigators wishing to design or modify chambers for the requirements of their own experiments.

Analytical Experiences with Beryllium Determinations in a Comprehensive Air Pollution Study*

SPENCER R. WATTS, B.S., FRANCIS X. WALSH, M.S., and VIRGINIA M. VOUGHT, B.S.

Occupational Health Laboratory, Pennsylvania Department of Health, Harrisburg, Pennsylvania

A STUDY to determine atmospheric concentrations of beryllium in the area of a beryllium plant located in Pennsylvania was recently undertaken by the Division of Occupational Health of the Pennsylvania Department of Health. The development of a routine laboratory procedure for the determination of beryllium was required for this study. This study was supported by a grant from the National Institutes of Health, U. S. Public Health Service.

As a method of determining the concentrations, twenty high volume type samplers were placed in the area surrounding the plant in a prescribed pattern (Figure 1). As a comparison measure between this and other communities, five background samplers were also placed at various locations in a highly industrial area in another part of the state. Two of these samplers were placed near a steel plant. All of the samplers were run for forty-eight hour periods and power for their operation was obtained from a variety of domestic sources. The filters were then removed from the samplers and mailed to our laboratory. Information such as weather conditions, wind directions, weight of particulate collected, and air volumes were recorded for each sample. The filter samples were then analyzed spectrographically[1] and results reported in terms of micrograms of beryllium per cubic meter of air. The air volume filtered was determined by recording initial and final sampler flow rates and multiplying the average of these two readings by the time of the sampling period. The resultant value was then converted to cubic meters.

All samplers were operated in as similar a manner as possible so that results, while not absolute, should be comparable on a relative basis. Our samplers (High Volume type) were calibrated using a Roots-Connersville type LP rotary gas displacement meter.

The samples were collected on 8 x 10 inch 1106-BH glass fibre filter web (Mine Safety Appliances CT-75428). This filter was selected for use because of its high collection efficiency,[2] ease of storing and mailing, relative strength of filter under varying sampling conditions, negligible hygroscopic qualities and fairly stable blank values for beryllium. Its flat rectangular shape facilitated cutting a 10 per cent section of the filter for analysis. The rest of the filter was then saved for reanalysis if necessary. Prior to the study, blank determinations were made on several different types of filters among them being three types of 6½ x 6½ inch FIXT-FLO Air Sampler Filter Sheet (Mine Safety Appliances, Pittsburgh, Pennsylvania) and two types of FIXT-FLO Pleated Air Sampler Filter Formed Filters 3¾ inches in diameter (Mine Safety Appliances) and Type AA 41 mm Millipore Filters, white grid (Millipore Filter Corp., Watertown, Mass.). The fairly high level of beryllium in the glass fibre filters was not desirable, but, in the light of the other advantages, this filter was selected.

In addition to determining beryllium in the atmospheric samples, this laboratory analyzed a number of lung tissue samples and a few soil samples. In most cases of tissue analysis, the samples submitted from the area under study showed slightly higher concentrations of beryllium than those from a background area. These results will be reported by Lieben and Metzner[3] at a later date. Although we do not have enough data on soil samples at this time, there seems to be a trend toward a similar correlation.

A literature review of colorimetric, spectrographic and fluorometric techniques was undertaken to determine which method would be most suitable for our purposes. The spectrographic method was chosen because of its reasonable sensitivity and accuracy, and a specific indication of beryllium could be obtained.

Sample preparation and actual analytical determination follow, in general, the procedure

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, April 30, 1959.

demonstrated by Cholak and Hubbard.[3, 4] We have, in the course of our daily routine, made minor changes in procedure which facilitate the analysis of the large number of samples submitted to our laboratory. Our experience with the cathode layer method of excitation did not prove fruitful or convenient. The critical alignment necessary required considerable readjustment and time to attain acceptable results. Further, the range of beryllium concentrations in atmospheric sample filters did not require extreme sensitivity.

Analytical Procedure

Filters are numbered and weighed before being sent to sampling stations. Samples are returned to the laboratory by mail in 4½ x 10½ inch brown paper envelopes in which the filters have been folded in half lengthwise inside of a light cardboard folder. Each filter is weighed to a constant weight after being exposed to room temperature and humidity for about ten minutes. A 2½ x 2½ inch square which represents one-tenth of the total sample area is cut and used for analysis. Unused portions of the filter are returned to mailing envelope and filed for occasional random reanalysis. The samples are treated with hydrofluoric and sulfuric acid to volatilize the glass and wet-oxidized by the addition of nitric and perchloric acids. The insoluble sulfates are separated by centrifuging from the acid solution and beryllium isolated by precipitation with ammonium hydroxide in the presence of a phosphate solution. The resulting precipitate is dissolved in hydrochloric acid and extracted with cupferron to remove iron. At this point, because of the wide range of concentrations encountered in field samples, it is obvious that dilutions are frequently necessary to bring the concentration within the working limits of the procedure. The dilution factors are obtained by estimating the concentration of beryllium. These estimates are based on previous results obtained from the same stations under similar conditions. By this technique we were able to place about 70 per cent of the samples within the working limits on the first dilution.

Sample solutions are combined with the internal standard solution and concentrated to one milliliter. Duplicate electrodes are loaded with 0.2 ml portions, and analyzed spectrographically. Final calculations are obtained as follows:

$$\frac{10[(A \times B) - C]}{V}$$

= Micrograms of beryllium per cubic meter of air,

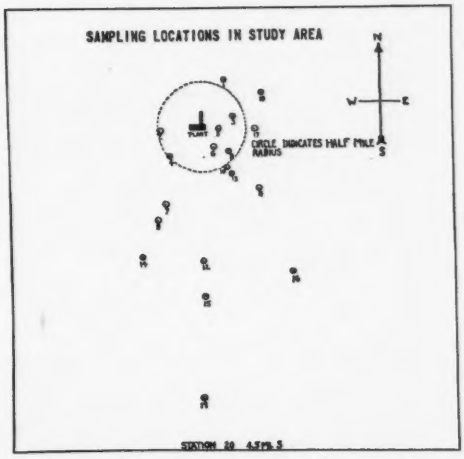

FIGURE 1. Showing sampling locations relative to the plant. Scale ½ inch = 1.0 mile.

where

A = micrograms of beryllium per final one ml volume,
B = aliquoting factor,
C = average micrograms of beryllium per 2½-inch x 2½-inch square of blank filter,
V = air volume in cubic meters.

Since the size of the square of the filter cut for analysis represents 10% of the total sampling area, we multiply by ten to obtain total net filter concentrations.

As of May, 1959, more than 750 samples have been analyzed. As will be discussed later, this determination is a long procedure and chemical preparations prior to spectrographic analysis are most time-consuming. A most important step is the preparation and acid cleaning of glassware.[5] All glassware must be washed first in soap and water, then cleaned with acid and rinsed completely with double-distilled water to safeguard against beryllium contamination.

Standardization

Our standard curve was derived from results obtained by adding known amounts of beryllium in solution (stock solution $BeSO_4 \cdot 4H_2O$ in 2 per cent HCl) to the prescribed amount of internal standard (thallium and buffer salts) and adjusting the total volume to one ml. Duplicate electrodes were loaded with 0.2 ml of solution in the same manner as the samples and analyzed spectrographically. The workable range of concentrations was 0.05 to 1.0 microgram of beryl-

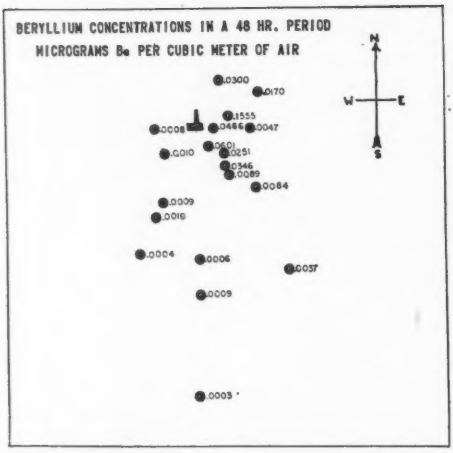

FIGURE 2. Showing beryllium concentrations at various locations. Scale ½ inch = 1.0 mile.

TABLE I

Typical Recovery Values for Beryllium Added to Blank Filter Squares

(Expressed as micrograms of beryllium)

Added	Found
0.050	0.045
0.100	0.078
0.500	0.498
1.00	0.91

TABLE II

Variation of Concentrations at Station No. 6, during Length of Study

(Twenty-five samples analyzed)

Micrograms beryllium per cubic meter of air		
1. 0.0633	9. 0.0516	18. 0.0116
2. 0.0350	10. 0.0616	19. 0.0435
3. 0.0526	11. 0.0601	20. 0.0371
4. 0.1422	12. 0.0084	21. 0.0114
5. 0.0235	13. 0.0018	22. 0.0456
6. 0.0606	14. 0.0137	23. 0.0695
7. 0.0302	15. 0.0035	24. 0.0359
8. 0.0072	16. 0.0044	25. 0.0232
	17. 0.0026	

Mean—0.0360
Median—0.0350
Standard deviation—0.0307

lium per milliliter of final volume. Recently we have begun to run four or five standards on each spectrographic plate so that any changes in the working curve become immediately recognizable. As a check on the recovery of the procedure, standard beryllium solutions were added to 2½ x 2½ inch squares of blank glass fibre filters and these were dried and treated the same as samples. See Table I for results.

Quality Control of Analytical Results

To eliminate regular anticipation of results in the laboratory, occasional samples were submitted as if they had been collected in the area under study when actually they had been collected in a background area.

Another set of samples was submitted from both a control area and the area under study with no information other than air volume. This made any anticipation of beryllium levels impossible and also necessitated more work to attain results. However, concentrations were determined which compared well with previous results.

From each lot of glass fibre filters several determinations were made to find the beryllium content of the blank filters. Results of these determinations were averaged and the values used as a base line for that particular lot. The average variation within one lot of blank filters is 20 per cent of the amount present based upon the duplicate analysis of three filters randomly selected from each lot. The concentration of beryllium in blank filters can be as high as 2.5 micrograms per filter.

Analytical Results

Although the working curve possessed a limited range, the actual concentrations encountered were as low as 0.0001 microgram of beryllium

TABLE III

Concentration Changes before, during and after Vacation Period at Ten Stations in Study Area

(Micrograms beryllium per cubic meter of air)

Sampling Station No.	Before vacation 7/15 to 7/17**	During vacation 7/23 to 7/25**	After vacation 7/27 to 7/29**	After vacation 8/26 to 8/28**
1	.0199	.0006	.0397	.0450
2	.0091	.0006	.0013	.0457
3	.0503	.0015	.0309	—*
4	.0040	.0009	.0009	.1292
5	.3009	.0081	.0446	.0614
6	.0018	.0035	.0114	.1468
7	.0022	.0009	.0003	.0773
8	.0268	.0028	.0055	.1036
9	.0009	.0004	.0002	.0800
10	.0129	.0006	.0008	.1279

* Sample lost due to motor failure.
** Sampling Period.

per cubic meter in a background area, to as high as 0.300 microgram of beryllium per cubic meter in a few samples taken near the plant. By examination of the concentration values, a general pattern became apparent. The pattern consists of high concentrations in areas close to the plant with reduced concentrations as distance from the plant increases. The concentrations also appear to be influenced by local meteorological conditions. A typical set of results has been superimposed on the sampling station map and is shown in Figure 2. There is considerable variation in concentrations within one sampling station, however, the results are still within the general pattern. Table II demonstrates the variety of concentrations found in a single station for 25 different sampling periods.

The comparison of data during production and vacation down-time offers an interesting point. Concentrations of beryllium during plant vacation periods are for the most part, significantly lower than those found during normal production periods. In Table III we have tabulated results from ten of the sampling stations for samples taken at four different periods surrounding the vacation time of the plant. It can be noted that there are variations within these results which can be attributed to the surrounding topography and meteorological conditions. These values alone should not be interpreted as a complete indication of the concentration changes. A full description of this area and the air pollution aspects may be found in the study by Sussman et al.[7]

The average concentration of beryllium found in five sampling stations located in the background area was 0.0002 microgram per cubic meter.

Conclusion

In this description of laboratory experiences, we have presented techniques used in an extensive air pollution study of a specific area. This location was sampled using high-volume type samplers. The filter samples were analyzed for beryllium and the results are described and discussed. The laboratory efforts were directed toward determining total beryllium concentrations with no attempt to identify any beryllium compound. It is suggested that studies in this direction merit investigation. Also suggested are studies for other elements which could be identified with other local sources.

Acknowledgment

The assistance of P. M. Fairlamb and L. T. Gonshor in performing much of the analytical work is gratefully acknowledged.

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Space Age Chemistry

A SYMPOSIUM ON Space Age Chemistry was presented by the Cincinnati Section of the American Chemical Society on October 29, 1959. Several speakers discussed high energy oxidizers and fuels now in use and proposed. Of particular interest was the role that polymers are playing in a wide range of applications in rocket construction and as components of solid fuels. New and unusual bonding systems are being investigated to obtain polymers stable in anticipated conditions. Phosphinoborines, triazines, and aluminoxanes have been prepared while aluminum-phosphorus, tin-silicon, boron-phosphorus, and others have been proposed. In the search for suitable refractory metal alloys, molybdenum-niobium alloys are of greatest interest. Tungsten alloys will probably be developed whereas chromium and vanadium alloys may be retarded by technical deficiencies of application. Of the rare elements, rhenium appears to be of special value.

An Air Pollution Study of a Community Surrounding a Beryllium Plant

VICTOR H. SUSSMAN, JAN LIEBEN, M.D., and JOHN G. CLELAND

Division of Occupational Health, Pennsylvania Department of Health, Harrisburg, Pennsylvania

Introduction

A LARGE portion of the beryllium metal and its alloys produced in the U. S. is manufactured in a plant located in Pennsylvania. There has been some concern in Pennsylvania with human exposure to beryllium for a number of years.[1] The Division of Occupational Health of the Pennsylvania Department of Health has conducted extensive studies of industrial exposure[2] and in 1955 directed attention to the study of possible exposures to citizens of the community surrounding the plant. A plan for an epidemiological and engineering study of the community was developed and, under a grant from the National Institutes of Health, U. S. Public Health Service, field work was started in May, 1958. The preliminary epidemiological findings have been reported in detail by Lieben, et al.[3] and show that, as of March, 1959, more than twenty cases of non-occupational chronic beryllium disease had been reported in the community under study. Beryllium disease was a primary or secondary cause of death in nine of these reported cases. The purpose of this paper is to report on the initial findings of the engineering phase of this study.

Plant Operations

The plant under study occupies approximately fifteen acres, much of which is devoted to the storage of beryl ore. Approximately three hundred people are employed in production operations. There are two or sometimes three shifts daily and operations are carried out on a six and sometimes seven-day week schedule.

Raw crystalline beryl ore is refined to beryllium oxide from which beryllium copper alloys, beryllium aluminum magnesium alloys, beryllium copper tools and some beryllium metal are produced. The ore is crushed, ball milled and agitated to form a sludge which is mudded with sodium silico-fluoride and then briquetted. The briquette in turn is sintered, crushed, milled, leeched, and filtered into cakes of beryllium hydroxide which is calcined to form beryllium oxide. The oxide is combined with metallic copper and carbon in an electrical arc furnace to form a master beryllium-copper alloy from which commercial beryllium alloys are made. Many of these alloys are founded at the plant into spark proof tools, or made into billets for hot or cold mill work, the end products of which are rods, strips, and wires.

The metal processes are typical of the metal industries in general. Billets are founded into tools which are subjected to trimming, grinding, and machining. They are also heated and flat rolled. Wires and rods are drawn or extruded, strips are annealed, bright-dipped, and in some instances plated. In the production of beryllium metal, the oxide is converted to a fluoride and then to the metal by magnesium reduction in an induction furnace. The plant has recently shifted a major portion of its production of beryllium metal to a newly built plant in another part of the state.

Sampling Methods

The community and plant under study are situated in a shallow valley. Nearby mountains to the east and south range between 800 and 1140 feet. Toward the northwest and west, the country is rolling, with nearby hills, seldom exceeding five hundred feet in elevation. The valley contains fertile farm land with a community, three to five miles south of the plant, known primarily as a manufacturing center.

The prevailing wind directions are from the northwest for nine months in the year, the other three months from the southeast. The yearly average hourly wind speed is between eight and ten miles per hour.

The locations of the neighborhood cases of non-occupational beryllium disease are described by Lieben and Metzner.[3] It should be noted, in any correlation made between the results of the engineering phase of the study and the location of the neighborhood cases, that the locations of these cases depend to a large extent upon the distribution of areas of dense population. The fact that most of the neighborhood cases appear to be downwind of the plant may have signifi-

cance but the community is not uniformly distributed around the plant.

The general atmospheric sampling program was started in May, 1958 with the operation of ten high volume samplers (General Metal Works, Cleves, Ohio). It was originally planned to locate the samplers at equidistant points on the circumferences of a number of concentric circles with the plant at the center. The northwest quadrant of the study area is sparsely populated and it was not possible to find many accessible sampler locations. In June, 1958, ten additional samplers were put into operation. The samplers were operated for 48-hour periods. Sampling was continuous except for short periods when only three 48-hour runs per week per sampler was made. Of the twenty samplers, nineteen were operated at ground level and one was placed on a building roof approximately fifty feet above ground level.

The high volume samplers were placed in standard vertical shelters as used by the National Air Sampling Network.[3] Suspended particulates were captured on 8″ x 10″ glass fiber filters (Mine Safety Appliances Company, Type 1106BH) and analyzed for beryllium with the use of an emission spectrograph.[4]

Much of previous out-of-plant monitoring for beryllium has been performed using Whatman #41 filter paper and the M.S.A. Type S unimpregnated filter.[5, 6] The 1106-BH Glass Fibre Filter Web was chosen for this sampling program for the reasons listed in reference[4] and because it has the highest filtering efficiency of the filter media available for this type of sampling.[7]

The high volume samplers were found to operate satisfactorily on 48-hour cycles. Between 4000 and 5000 cubic meters of air were filtered during the 48-hour sampling period. There was no evidence that the motors were heating excessively after 48-hour runs sampling concentrations of suspended particulates of the order of 100 μg per cubic meter. It was necessary to change brushes approximately every five hundred hours. The average motor life was 1500 hours, but it was found that a few motors lasted only 1000 hours. The samplers were calibrated with the use of a direct displacement pump (Roots-Connersville, 2½ x 7 type LP, Rotary Displacement Gas Meter) and at times with the use of a calibrated orifice. It was found that there was no significant change in the samplers' calibration curve after brush changes, motor inspections, or even after cleaning flowmeters. A spherical sapphire float in the flowmeter proved to be more rugged and easier to read than the usually used pointed plastic float. It was found that it was necessary to recalibrate samplers after replacing motors. Calibration of samplers in the field using the calibrated orifice was found to be accurate, to the nearest cfm.

Atmospheric Beryllium Concentrations

The cumulative frequency distribution of beryllium concentrations found in the area of the plant and at background stations is shown on Figure 1. During a four-month period (May through August, 1958) five hundred 48-hour samples were obtained from the samplers located in the area of the plant. The mean concentration of beryllium found in the area of the plant was 0.0155 μg per cubic meter of air (STP). The median concentration was 0.0040 μg per cubic meter with a standard geometric deviation of 5.5. Ten per cent of the samples showed concentrations in excess of 0.035 μg per cubic meter. The reasons for the skewness of this distribution will become obvious during the discussion of Figure 2.

During the same four-month period, five samplers were operated in two communities in a different area of the state. Two of these samplers were placed in the area of a large steel plant. The mean concentration of eighty samples obtained from the five background samplers was 0.0002 μg per cubic meter, the median concentration was also 0.0002 μg per cubic meter. The standard geometric deviation was 2.1, indicating a relatively uniform distribution, which is to be expected in background sampling.

The National Air Sampling Network reports[8,9] that no detectable quantities of beryllium have been found at most of their sampling sites, and where detectable amounts have been found, concentrations are of the same order of magnitude as those found at the background stations of this survey.

Figure 2 is a group of concentration roses representing a correlation between concentration and wind direction at the various sampling sites. The roses were obtained by weighting concentrations with daily prevailing wind directions. For example, the bar pointing towards the west, in the rose for station 5, indicates that an average concentration of approximately 0.11 μg per cubic meter was measured at station 5 when the prevailing winds were from the west. The decrease in atmospheric concentrations of beryllium with increasing distances from the plant is obvious (note that the concentration scale is the same for all twenty stations). Figure 3 tabulates the average concentrations found at various distances and directions from the plant.

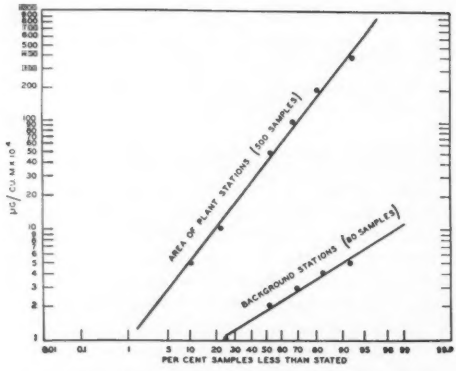

FIGURE 1. Cumulative frequency distribution of beryllium concentrations at sampling stations in the plant area and in the background area.

The directions in Figure 3 refer to the location of the samplers with respect to the plant and not prevailing winds.

The concentration roses prepared upon completion of this study will be based upon hourly prevailing wind directions. This requires the handling of a considerable quantity of data. A system for compiling and reducing these data is now being developed. The presentation of data based on daily prevailing winds, (as in Figure 2) is adequate for preliminary evaluations.

A meteorological field station (Beckman and Whitley Model 170 Wind Speed and Direction Recorder) was erected approximately one mile from the plant and about three miles north of a local U. S. Weather Bureau Station. Wind speed and direction readings obtained at both stations were compared showing that there were no unusual wind movements occurring in the area. The data from the two stations coincided and it was possible to use the tabulated U. S. Weather Bureau data and dismantle our station.

During the four-month period in which these samples were obtained, the plant was closed down for three weeks for a vacation. All plant operations were discontinued for two weeks of this period and partial operations were conducted during the third week. The cumulative frequency distributions of beryllium concentrations found, during plant operation and while

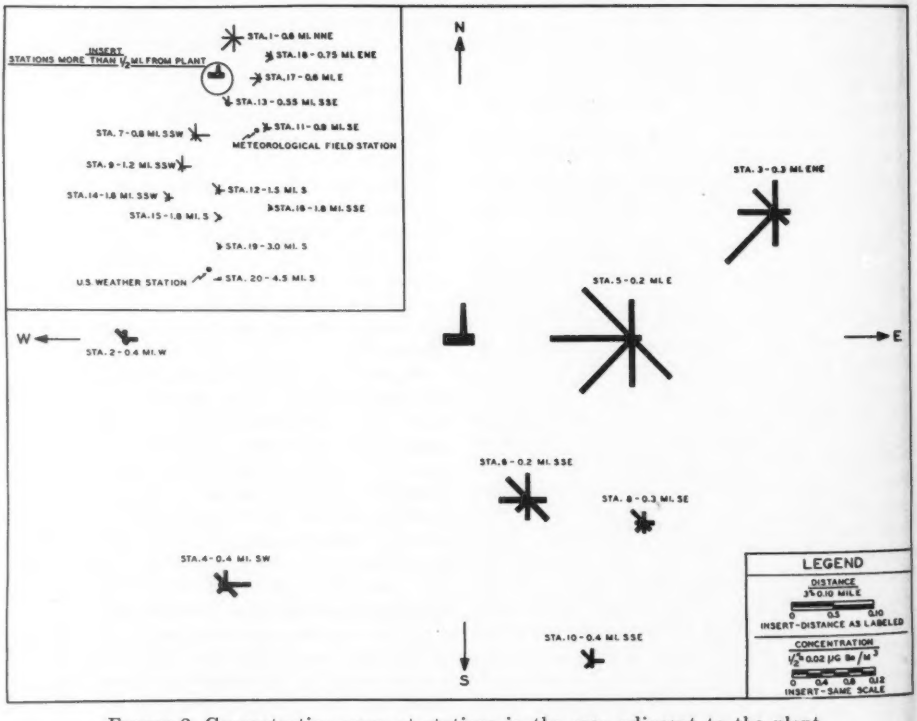
INSERT
STATIONS MORE THAN ½ MI. FROM PLANT
STA. 1 - 0.6 MI. NNE
STA. 18 - 0.75 MI. ENE
STA. 17 - 0.6 MI. E
STA. 13 - 0.55 MI. SSE
STA. 7 - 0.8 MI. SSW
STA. 11 - 0.8 MI. SE
METEOROLOGICAL FIELD STATION
STA. 9 - 1.2 MI. SSW
STA. 14 - 1.8 MI. SSW
STA. 12 - 1.5 MI. S
STA. 16 - 1.8 MI. SSE
STA. 15 - 1.8 MI. S
STA. 19 - 3.0 MI. S
U.S. WEATHER STATION
STA. 20 - 4.5 MI. S
N
W
E
S
STA. 2 - 0.4 MI. W
STA. 3 - 0.3 MI. ENE
STA. 5 - 0.2 MI. E
STA. 6 - 0.2 MI. SSE
STA. 8 - 0.3 MI. SE
STA. 4 - 0.4 MI. SW
STA. 10 - 0.4 MI. SSE
LEGEND
DISTANCE
INSERT - DISTANCE AS LABELED
CONCENTRATION
INSERT - SAME SCALE

FIGURE 2. Concentration roses at stations in the area adjacent to the plant.

DIRECTION	MILEAGE													
	.2	.3	.4	.5	.6	.75	.8	.9	1.2	1.5	1.6	1.8	3.0	4.5
N.E.					.0170									
E.N.E.		.0475				.0066								
EAST	.0827				.0090									
S.E.		.0166						.0054						
S.S.E.	.0360		.0117	.0062								.0037		
S.										.0082		.0055	.0017	.0011
S.S.W.							.0114		.0096		.0060			
S.W.			.0158											
WEST			.0085											

AVERAGE CONCENTRATIONS (ALL STATIONS) BY DISTANCE

0 TO .5 MILE	.6 TO 1.0 MILE	1.1 TO 2.0	> 2.0
0.0281	0.0107	0.0066	.0014

FIGURE 3. Average concentrations at the various sampling stations.

the plant was shutdown (including the one-week period of partial operation), are shown in Figure 4. The mean concentration during the period of plant operation was 0.0190 μg per cubic meter, the median concentration was 0.0051 μg per cubic meter and the standard geometric deviation of the samples was five. During the period of plant shutdown, the mean concentration was 0.0026 μg per cubic meter, the median concentration was 0.0010 μg per cubic meter with a standard geometric deviation of 3.3. The comparatively high concentrations, as compared to background concentrations, found during the period of plant shutdown may be partially accounted for by the fact that the plant was in semi-operation for one week during the three-week shutdown period. The mean concentration during the two-week period of complete shutdown was 0.0015 μg per cubic meter, while the mean concentration during the one-week period of partial operation was 0.0047 μg per cubic meter. There is also a possibility that beryllium deposited on the ground and other surfaces in the area of the plant may become airborne. To evaluate this premise, grab samples of top soil were taken at some of the sampling sites. These preliminary samples indicated decreasing concentrations of beryllium in the soil with increasing distances from the plant. Procedures are now being developed to conduct a soil sampling program in the area of the plant and other areas of the state. An indication of

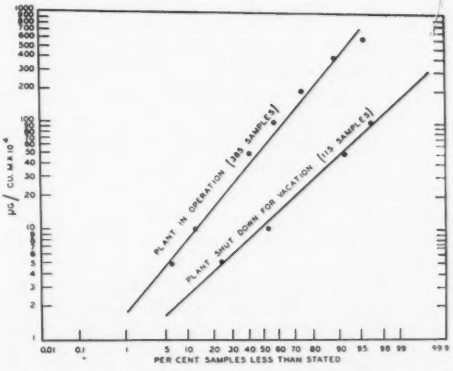

FIGURE 4. Cumulative frequency distribution of beryllium concentrations during operating and non-operating (vacation) periods of the plant.

the extent of ground pick-up may be obtained by weighting concentrations, as determined with the use of the high volume samplers, with wind speeds. This information is now being compiled. It is obvious, nevertheless, that a marked increase in atmospheric beryllium concentrations occurs when the plant is in operation as compared to a period of plant shutdown.

In May, 1959 the sampler locations will be changed, so that samplers can be located in areas in which neighborhood cases have recently been discovered. The revised arrangement of

samplers will also permit a more detailed study of the effect on atmospheric concentrations of wind directions and speeds. The samplers will be placed equidistant from each other within a rectangular area.

Discussion

A concentration of 0.01 μg per cubic meter has tentatively been established as a maximum atmospheric concentration for non-occupational 24-hour exposure.[10] The Atomic Energy Commission recommends that, "In the neighborhood of an AEC plant handling beryllium compounds, the average concentration at the breathing zone level should not exceed 0.01 μg per cubic meter."[6] It is further required that, "In the event that the maximum average neighborhood concentration at the ground during any calendar month, as determined on a monthly basis, is greater than 0.01 μg per cubic meter but less than 0.05 μg per cubic meter, the plant will be expected to inform the AEC of specific procedures which will be undertaken to reduce the airborne concentration," A previous study of non-occupational berylliosis[5] recommended that, "The average daily concentrations of beryllium in the vicinity of a plant producing and processing beryllium should be limited to 0.01 μg per cubic meter." According to Silverman,[11] "The present non-occupational permissible concentration for beryllium exposure in the environs can be attained with reasonably economic methods." The data contained in this preliminary report are sufficient to require that additional controls be employed by the plant so as to reduce atmospheric concentrations of beryllium in the vicinity of the plant.

In order to make specific recommendations for control and obtain a basis for the determination of the degree of control attained, a stack sampling program will be conducted at the plant. It is hoped that the results of the stack tests correlated with information on the installation dates of collectors and production rates, can be used to estimate atmospheric concentrations that may have occurred in the past.

The high volume sampler proved to be an efficient and practical tool for specific source sampling. The data obtained using this sampler were reduced to determine the location of the source and the relationship of atmospheric concentrations to meteorological variables.

There is a need for coordinated epidemiological and engineering studies to provide basic information for air pollution control. Studies of this type aid in the establishment and revision of non-occupational threshold limits, and establish a basis for requiring control. The development of sampling techniques and methods of data evaluation are important in providing standards for control requirements and specifying areas in which emission standards are to be applied.

Acknowledgments

The authors wish to acknowledge the valuable assistance of R. C. Wanta, U. S. Weather Bureau, and personnel of the Public Health Service, U. S. Department of Health, Education, and Welfare.

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Use of a Gas Chamber for Testing Air Samplers*

R. O. McCALDIN, Ph.D., and E. R. HENDRICKSON, Ph.D.†

U. S. Public Health Service, Robert A. Taft Sanitary Engineering Center, Cincinnati, Ohio

Introduction

THIS PAPER describes a gas chamber used to test air sampling equipment, and it reports on the collection efficiency and operating characteristics of three types of air samplers. One is a commercially available scrubber, another a sampler of special design, and the third a midget impinger.

Gas Test Chamber

Gas test chambers can be operated either statically or dynamically. A static chamber is one in which a measured quantity of test gas is admitted to the chamber and a known initial concentration is established. When samples are withdrawn from the chamber, fresh make-up air is added to replace the gas mixture removed, and there is a gradually decreasing gas concentration in the chamber. A dynamic chamber is one in which a suitable gas mixture is continuously passed through the chamber.

The gas test chamber used in this work is located in the Sanitary Engineering Laboratory at the University of Florida.[1] It was designed for both static and dynamic operation. However, in these tests it was used only under static conditions. The chamber is of wood frame construction, and has an interior of pressed-wood hardboard coated with polyvinyl chloride paint. The chamber is a cube with six-foot sides and has a volume of 216 cubic feet (6.12 cubic meters). It is equipped with a number of sampling ports and a fan for mixing air and test gas. This fan was mounted on a shaft extending through the chamber roof. The motor was outside the chamber to avoid possible air contamination from motor lubricants and to minimize the exposed surface of the chamber. A door comprises one face of the chamber and is held in place with C-clamps. A plastic coated gasket prevents gas leakage at the door.

This chamber underwent a number of tests to determine its ability to retain a given concentration of various test gases. The first test was to determine leakage from the chamber under different operating conditions. This was done by adding a measured volume of air to the chamber and then continuously determining its subsequent pressure. For normal operating conditions, near-atmospheric pressure in the chamber (±0.2 inches of water), the leakage was found to be about one per cent of the chamber volume per day.

Test gases were admitted to the chamber with a hypodermic syringe. Known volumes of gas could be admitted by this method, and as long as care was taken to let the gas temperature in the hypodermic syringe adjust to a known value, the weight of gas admitted could be readily calculated by using the ideal gas law. In turn a theoretical gas concentration could be calculated for the chamber.

It was found that, after a measured quantity of gas was injected and mixed in the chamber by the fan, the theoretical gas concentration was not achieved. The experimental concentration was always less than the theoretical concentration by a uniform percentage. The reduction in experimental concentration could be due to adsorption of the test gas on walls of the chamber. This possibility was evaluated by determining the effect of additional chamber surface on gas concentration. A number of large cardboard panels, coated with polyvinyl chloride similar to the interior surface of the chamber, were hung in the chamber to increase the total surface by approximately one-half. The increased chamber surface was found to reduce further the experimental gas concentration, the reduction being proportional to the increased surface of the chamber. On the basis of this test the difference between the theoretical gas concentration and that actually achieved appears to be due to adsorption of gas on the chamber surface. Experimental findings fit the empirical Fruendlich adsorption equation:[2]

$$\frac{x}{m} = kp$$

where:

x = quantity of gas adsorbed
m = surface area
k = constant
p = partial pressure of gas.

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association in Chicago, Illinois, on April 30, 1959.

† Dr. Hendrickson is Professor of Sanitary Engineering at the University of Florida, Gainesville, Florida.

It was found experimentally that gas adsorption on the chamber walls reduced the actual sulfur dioxide concentration from its theoretical value by approximately ten per cent. With hydrogen sulfide the reduction was about six per cent and with hydrogen fluoride about thirty-three per cent.

The ability of the chamber to retain a given concentration of gas over a period of time was also studied. It was found that sulfur dioxide concentrations diminished about seven per cent for each day of storage. Hydrogen sulfide diminished about twenty-six per cent per day and hydrogen fluoride about twelve per cent per hour. The tests were conducted with gas concentrations ranging from about 0.1 to 7.0 parts per million by volume, and loss rates were similar for all test concentrations used. It can be concluded that the chamber with its present interior surfacing is suitable for retention of sulfur dioxide and hydrogen sulfide. Hydrogen fluoride reacts so rapidly with the chamber surface that samplings of only short duration can be performed.

It was calculated and confirmed by test that in static operation the removal of a 200-liter sample from the chamber would reduce the gas concentration by approximately three per cent. The total sample taken in any given test was generally well under 200 liters, and consequently the dilution effects due to sample removal were not great enough to interfere with test results.

Test Methods

The gas samplers described below were evaluated with respect to their operating characteristics and collection efficiencies when sampling for sulfur dioxide in the 0.3 to 1.4 ppm range. The test chamber was used to furnish a uniform reservoir of the sulfur dioxide-air mixture. The usual test procedure was to inject a measured quantity of sulfur dioxide into the chamber, start the mixing fan, and after ten minutes of mixing time, withdraw samples. Samples were drawn through a variable area flowmeter, the sampler, a valve, and a pump connected in series. In field sampling one would normally place the sampler first in the collection train. In this work, however, the variable area flowmeter was placed ahead of the sampler in order to avoid the additional flow calibrations required each time a different sampler or flow rate produced a different pressure loss in the sampling train.

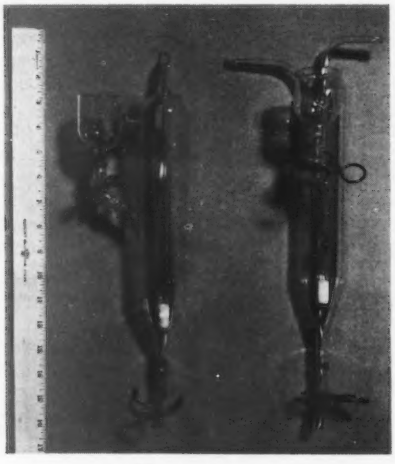

FIGURE 1. Hendrickson air sampler.

Analysis for sulfur dioxide was by the colorimetric method, described by West & Gaeke.[3] In this method the scrubbing solution, 0.1 molar sodium tetrachloromercurate, reacts with sulfur dioxide to form a relatively stable, nonvolatile solution of disulfitomercurate. The determination of disulfitomercurate is based on the red-violet color developed when an organic dye (pararosaniline hydrochloride-hydrochloric acid and formaldehlyde) is added to the solution. Samples are analyzed colorimetrically, and values are read from a standard curve.

Description of Samplers

Hendrickson Sampler

The Hendrickson sampler (Figure 1) has a cylindrical glass frit which is built into a container for holding the scrubbing solution. The side arm cup allows for ready addition of sampling solution, and the cone-shaped bottom of the container allows for rapid and complete removal of sample. This device is ideally suited for laboratory work, but the amount of glassware appended to it makes it rather delicate, and requires careful handling for field work.

This sampler requires a minimum of 75 ml of solution to keep the fritted bubbler submerged and is normally operated with 100 ml of scrubbing solution. The maximum flow rate is determined by the maximum rise of bubbles in the sampler without loss of absorbing solution. This flow was found to be around twenty liters per minute (0.7 cfm) but varied with the frothing tendency of the scrubbing agent. It was found that sodium tetrachloromercurate had much less tendency to froth when stored in a glass container, as compared to the same solution stored in some types of plastic containers.

Coarse glass frits used in the Hendrickson

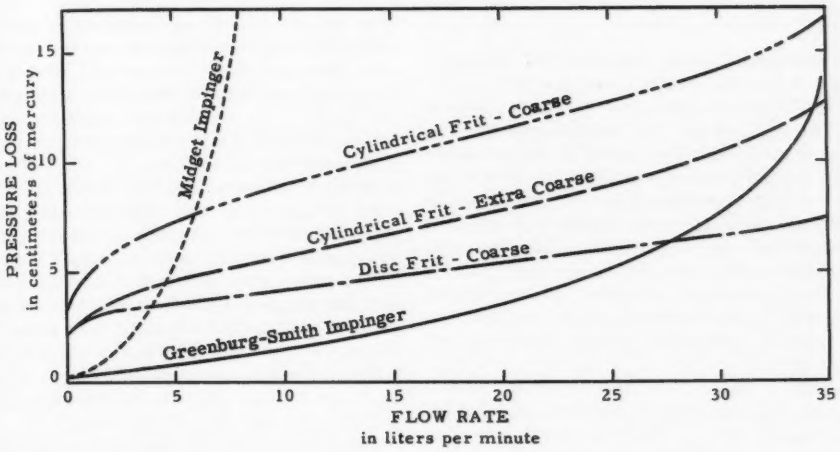

FIGURE 2. Relationship between pressure loss and air flow rate through different types of bubblers.

sampler have pores ranging from 90 to 150 microns in size. Due to variations among the individual frits the pressure loss characteristics as related to air flow is a bit different for each sampler. An average sampler of this type causes a pressure drop equal to about three cm of mercury before air will bubble through the frit, and it takes about eleven and one-half cm of mercury to achieve a maximum flow rate of twenty liters per minute (0.7 cfm). It can be seen in Figure 2 that the head loss through this coarse, cylindrical frit, bubbler is greater than that for the other types of bubblers used in this study.

Laboratory Gas Washing Bottle

The laboratory gas washing bottle is a standard item listed in most of the glassware catalogs. It is composed of a cylindrical container with a fixed base. The top contains the probe and fritted disc. The two pieces are matched and have a good seal at the ground glass joint. This sampler is fairly rugged and easy to use with the exception that it takes a little patience to empty the sampler of scrubbing solution and remove the last portion of solution trapped below the fritted disc.

Fifty milliliters of scrubbing solution will amply cover the fritted disc, and 100 milliliters is commonly used with this device. The maximum sampling rate for this instrument is about 35 liters per minute (1.2 cfm).

The resistance to air flow for this type of sampler was found to vary from one sampler to the next due to porosity of the different frits. For an average sampler it takes a pressure drop equivalent to about 2.5 cm of mercury to start air flow. There is a pressure drop about equal to 7.3 cm of mercury associated with a maximum flow rate of 35 liters per minute (1.3 cfm) through the coarse disc frit used in this sampler. This is shown in Figure 2.

Midget Impinger

The midget impinger[4] is the most readily portable of the three samplers considered. The model used in these tests consists of a cylindrical container with volumetric graduations on the side, with the impinger probe mounted in a rubber stopper used to seal the sampler. This model has guide arms near the bottom of the impinger probe to insure that the probe is centered in the washing bottle. The midget impinger is compact and can be readily fitted into a sampling kit for field use.

This sampler has been designed for use with ten milliliters of scrubbing reagent. The economy in scrubbing reagent is apparent when compared with the quantity used by the larger samplers. Because of the small amount of solution used, accurate measurement of the scrubbing reagent is required.

The design flow rate for this sampler is 2.8 liters per minute (0.1 cfm). The maximum flow rate that can be achieved without loss of solution is about 3.9 liters per minute (0.14 cfm). As shown in Figure 2 the resistance to air flow for this type of sampler was found to be under 3.5 cm of mercury for its entire useable flow range.

Reproducibility of Results

It is difficult to give a figure for the reproducibility of sample results based on the sampler alone. The reproducibility of results depends on the reliability of gas flow measurements and of the analytical technique, as well as the reliability of the sampling instrument itself. In order to obtain comparable results for the three types of samplers tested, the same methods of gas flow measurement and analytical techniques were used. A variable area flowmeter was used for measuring flow rate; sampling time was measured with a stop watch; and the analysis was performed in a consistent manner throughout the tests.

A number of tests were run in which replicate samples were taken with each type of sampler. The average sulfur dioxide concentration was determined for each set of samples. The deviation of individual samples from the average in each test was then determined, and the average deviation for the entire set of data for each sampler was calculated. It was found that the average deviation for the laboratory gas washing bottle was three per cent. For both the midget impinger and the Hendrickson sampler the figure was five per cent.

The depth of the midget impinger probe in the collection medium was studied for possible effect on collection results. Samples were taken with the probe at the design distance of five millimeters above the base of the sampler and at approximately three and eight millimeters above the base of the sampler. These variations in probe depth did not produce any measurable difference in collection results. It should be emphasized that the impinger served in these tests as a gas washing bottle rather than a dust impinger for which it was designed.

In order to secure consistent collection results, it is important to have accurate measurement of gas flow and sampling time, careful rinsing of scrubbing reagent from the sampler and careful measurement of final sample volume. In the colorimetric analysis care is required in the control of reagent temperatures and time allowed for color development before readings.

Collection Efficiencies

The collection efficiency of a sampler can be determined in a number of ways. A method sometimes used is to arrange enough samplers in series so that the last sampler does not recover any of the test gas. It is then assumed that the entire quantity of gas is collected in the samplers. The amount collected in the first sampler in the series, compared with the total quantity of gas collected, represents the collection efficiency of the first sampler. This method is satisfactory only if collection efficiency does not decrease at the low gas concentrations to which the second and following samplers in the series are exposed.

Series testing was done with the three samplers used in this study. All three samplers gave a collection efficiency of greater than 99 per cent as reported by this series evaluation method. These tests were repeated for sulfur dioxide concentrations of 1.4, 0.6, and 0.3 ppm. Sampling rates for the midget impinger were varied from 1.0 to 3.9 liters per minute. For the laboratory sampler the flow rate was varied from 7.5 to 35 liters per minute. For the Hendrickson sampler the flow rate was 7.5 liters per minute.

Collection efficiency was also evaluated by comparing the gas concentration based on sampler results with the known gas concentration in the chamber from which the sample was taken. Results for the laboratory gas washing bottle and Hendrickson sampler were in general agreement with the predicted gas concentration in the chamber and with the results of series testing. A surprising result was disclosed for the midget impinger, however. It appeared that the gas concentration determined by the midget impinger was one-fourth to one-third lower than that predicted for the chamber and disclosed by the other two samplers. When this test was first performed, a fresh piece of tygon tubing had been used to feed gas from the chamber to the impinger. This test was repeated a number of times. After the tygon tubing had been exposed to sulfur dioxide gas for a number of days this anomaly in the impinger results disappeared, and comparable results were achieved between each sampler and the predicted chamber concentration. This test method showed that *fresh*

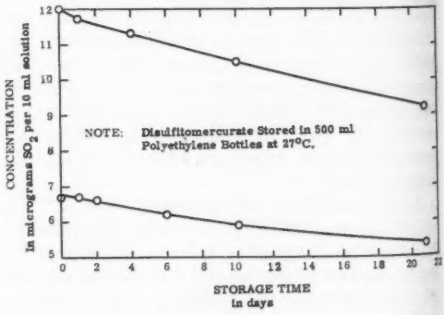
CONCENTRATION
In micrograms SO_2 per 10 ml solution
NOTE: Disulfitomercurate Stored in 500 ml Polyethylene Bottles at 27°C.
STORAGE TIME
in days

FIGURE 3. Effect of storage on the concentration of SO_2 found in samples.

tygon tubing in the sampling train can substantially reduce the amount of sulfur dioxide that reaches the sampler. This presumably reflects a chemical reaction between the tygon tubing and sulfur dioxide.

Testing of samplers from a known gas concentration has the advantage of allowing a check of over-all results. Not only is it possible to check the sampler collection efficiency, but any gas loss, such as that from the fresh tygon tubing, or metering error in the sampling system will also be disclosed. The sensitivity of this method is limited by the ability to predict the chamber gas concentration and by the variability in sampling results. Small errors or differences in collection efficiencey may be obscured by variation in experimental results. However, this technique readily discloses differences of five per cent or more.

Effect of Storage on Sample Concentration

Quite frequently field samples are taken at such distance from the laboratory that it may be necessary to store them for a time before analysis. Although the collected sulfur dioxide forms a relatively stable solution of disulfitomercurate, it appeared desirable to know what loss, if any, would occur in samples that were stored.

The effects of storage on sample strength were determined experimentally. A large sample of sulfur dioxide was collected in the scrubbing reagent. This was thoroughly mixed, and equal portions were poured into a number of 500-ml polyethylene bottles. The bottles were capped and stored for periods up to 21 days before the contents were analyzed. It was found that the strength of the solution decreased by one per cent per day of storage. The results of these tests are shown in Figure 3. This loss would not be significant for samples analyzed within a day or so of their collection. However, it becomes important when samples are collected at isolated field stations and stored for a week or more before being sent to the laboratory for analysis.

Summary

A test chamber has been described which offers a convenient gas reservoir for use in evaluating air sampling instruments. The chamber was evaluated with respect to its ability to retain different gases, and results are reported for the effects of leakage, surface losses, and decay rates on test concentrations.

Three types of air samplers were used to collect sulfur dioxide from the gas test chamber. For normal operating conditions of flow rate, and at sulfur dioxide test concentrations of 0.3 to 1.4 ppm, collection efficiency of the samplers is nearly 100 per cent. Under carefully controlled conditions it is possible to achieve reproducibility of sampling results with an average variance under five per cent for the three types of samplers tested. The accuracy of sample results depends on the care with which each sampling step is carried out. For best results consideration should be given to: composition of inlet tubing so that it is non-reactive with materials collected; elimination of any leaks in sampling train; accurate measure of gas flows; careful rinse of sampler and glass frits; accurate measure of scrubbing reagent; prompt analysis of sample, or due allowance for effects of aging on sample; and carefully controlled analytical techniques.

Regardless of the specific sampling system in use, it is advisable to ascertain its accuracy and reproducibility. One can then determine what reliance to place on findings and can determine the degree of significance to be placed on quantitative results.

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HYGIENIC GUIDE SERIES

Hydrogen Selenide

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 0.05 parts of gas per million parts of air, by volume (ppm).[1]

(1) *Basis for Recommendation:* Animal studies[2, 3] and clinical observations.[4]

B. SEVERITY OF HAZARDS:

(1) *Health:* High for both acute and chronic exposures.[2, 3, 4] Acute effects consist of eye, nose and throat irritation, and may be followed by acute pneumonitis. Subacute and chronic effects include garlic odor of breath, nausea, vomiting, metallic taste in mouth, dizziness, extreme lassitude and fatigueability. Even brief exposures produced liver damage in animals as well as weight loss and general debility.

(2) *Fire:* Gas will burn if ignited.

C. SHORT EXPOSURE TOLERANCE: 0.3 ppm (1 mg H_2Se/M^3) killed 50 per cent of guinea pigs[3] in a single 8-hour exposure. A concentration of less than 0.2 ppm for a maximum exposure of one month was reported by Buchan[4] to have produced signs and symptoms in man.

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: 0.3 ppm (1 mg/M^3) is considered by Dudley and Miller[3] to be dangerous for four hours' exposure. This concentration is not irritating and the odor perception of it is soon lost; therefore, if the odor of H_2Se is detected, the concentration is to be considered dangerously high.

II. Significant Properties

Hydrogen selenide is a colorless gas with a very disagreeable odor.

Chemical formula:	H_2Se
Molecular weight:	80.98
Relative vapor density:	2.8 (air = 1)
Melting point:	−64°C
Boiling point:	−42°C
Solubility:	270 ml soluble in 100 ml water at 22.5°C. More soluble in alkaline solutions. Soluble in carbon disulfide.

At 25°C and 760 mm Hg:

1 mg H_2Se/M^3	0.3 ppm
1 ppm H_2Se	3.3 mg/M^3

III. Industrial Hygiene Practice

A. RECOGNITION: Hydorgen selenide has no industrial uses but it may be produced by the reaction of acids or water with metal selenides (such as digestion of selenium-bearing ores of other metals) or by reactions which result in the generation of hydrogen in the presence of selenium. Since there have been a number of instances of unexpected generation of hydrogen selenide, this possibility should always be recognized in chemical reactions involving selenium metal or compounds.

B. EVALUATION OF EXPOSURE:

(1) *Instrumentation:* None

(2) *Chemical:* Absorption of the gas on soda lime with subsequent conversion to and estimation of elemental Se,[3] also absorption in hydrobromic acid–bromine solutions with final determination by Chernyi's method.[7]

C. RECOMMENDED CONTROL PROCEDURES: Avoid production of H_2Se during handling or processing other selenium compounds. Where selenium is present in materials which must be treated with acids, adequate ventilation should be provided to remove any gas formation. Do not permit workers to remain even for brief periods in areas where H_2Se is detectable by odor.

IV. Specific Procedures

A. First aid: Remove from exposure, place at bed rest immediately and call a physician.

B. Special medical procedures: Garlic odor on breath may be present after long or repeated exposures to low concentrations. It may be absent, however, in acute, high level exposures. Determination of urinary Se may be helpful; normal urinary values for unexposed males have been given as 0–15 micrograms with an average of 4–5 micrograms/100 ml.[6] The 24-hour excretion seems independent of urinary volume. In view of the toxicologic properties of hydrogen selenide, the physician may wish to review carefully the placement of persons with chronic liver or pulmonary disease.

V. References

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Antimony and Its Compounds

(Excepting Stibine)

I. Hygienic Standards

A. Recommended maximum atmospheric concentration (8 hours): 0.5 milligrams per cubic meter of air (mg/M³), calculated as antimony.[1]

(1) *Basis for Recommendation:* Animal experimentation and human experience.

B. Severity of hazards:

(1) *Health:* Moderate to high for both acute and chronic exposures, although systemic intoxication of occupational origin is uncommon. An apparent increase in heart abnormalities was found by Brieger[2] in persons exposed to antimony trisulfide, and heart injury has been demonstrated in experimental animals.[2, 3] According to Renes, upper respiratory irritation, pneumonitis, systemic effects (abdominal cramps, diarrhea, vomiting, dizziness, prostration) and dermatitis resulted from over-exposure to antimony oxide fumes contaminated with arsenic.[9] The acute intraperitoneal toxicity of antimony in rats decreased in the following order; antimony potassium tartrate, antimony metal, antimony trisulfide, antimony pentasulfide, antimony trioxide, and antimony pentoxide.[2] Acute intoxication can result from oral intake or therapeutic administration.[5] The single intraperitoneal LD_{50} doses for mice ranged from 14.4 to 46.4 mg/kg for 32 antimonials.[7] For rats, the intraperitoneal LD_{50} ranged from 10.1 to 4000 mg/kg, for antimony and five of its compounds.[2] Exposure of guinea pigs for several weeks to antimony trioxide dust at average concentrations of 45.4 mg/M³ produced pneumonitis, liver and blood changes, and a mortality rate of 16 per cent.[4] Dermatitis has been reported in persons manufacturing antimony trioxide.[8]

(2) *Fire:* Low. In common with many metal powders, finely divided antimony can present an explosion hazard.

C. Short exposure tolerance: Unknown for man.

D. Atmospheric concentration immediately hazardous to life: Unknown.

II. Significant Properties

Antimony is a silvery-white, soft metal having the following properties:

Chemical symbol:	Sb
Atomic weight:	121.76
Specific gravity:	6.69
Melting point:	630.5°C
Boiling point:	1380°C

III. Industrial Hygiene Practice

A. RECOGNITION: Mining and milling operations. The metal is used chiefly in alloys (bearings, lead storage batteries, type metal, Pewter). The compounds are used for flame proofing (trioxide), abrasives (resinoid grinding wheels—trisulfide), pigments, and drugs.

B. EVALUATION OF EXPOSURES:

(1) *Direct Instrumentation:* None available.

(2) *Chemical:* Collection of particulates by impingement, electrostatic precipitation, or mechanical filtration. Antimony determination by the rhodamine B method.[6, 9]

C. RECOMMENDED CONTROL PROCEDURES: Process ventilation should be used, as needed, to maintain atmospheric concentrations below 0.5 mg/M³ (calculated as Sb). Good personal sanitation must be practiced in order to avoid ingestion. Antimony and antimony compounds may generate stibine under reducing conditions. This possibility should be recognized and avoided.

IV. Specific Procedures

A. FIRST AID: No specific measures. Remove from exposure, place at bed rest, and call a physician in the event of acute symptoms during an exposure.

B. SPECIFIC MEDICAL PROCEDURES: Urinary excretion of antimony may be a useful indication of exposure.[8] Where there is likelihood of significant exposures, preplacement and periodic examinations should be conducted with particular attention directed towards the heart (including electrocardiographic examination), lungs, skin, and gastrointestinal system.

V. References

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Styrene Monomer

(Phenylethylene)

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 100 parts of vapor per million parts of air, by volume (ppm).[1]

(1) *Basis for Recommendation:* Human experience and animal studies.

B. SEVERITY OF HAZARDS:

(1) *Health:* Low, for both acute and chronic exposures. Capable of causing narcotic symptoms in man and animals but the odor and irritant effects of the vapor make voluntary exposure to narcotic concentrations unlikely. There have been no reports of serious illness from exposure to styrene vapors. Repeated skin contact may result in drying and irritation of the skin.

(2) *Fire:* Moderate when exposed to heat or flame. Flash point 88°F (closed cup). Explosive limits 1.1 per cent (29.3°C) to 6.1 per cent (65.2°C).

C. SHORT EXPOSURE TOLERANCE: 800 ppm inhaled by two human subjects for four hours produced immediate eye and throat irritation, and mild narcotic symptoms which persisted for a short period after termination of exposure.[2] Some persons engaged in operations at which concentrations were found to be 200–700 ppm complained of disagreeable odor and episides of nausea, loss of appetite and weakness of a few hours duration.[3]

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Unknown for man. This danger is limited by irritant properties of vapor. The maximum concentration achievable under ordinary room conditions, approximately 10,000 ppm, produced death in 100 per cent of rats and guinea pigs in three hours but there was 100 per cent survival after one hour exposure.[4]

II. Significant Properties

Oily, colorless liquid with a distinctive odor.

Chemical formula:	$C_6H_5CH{=}CH_2$
Molecular weight:	104.14
Boiling point:	145°C
Vapor pressure:	10 mm of Hg at 30.8°C
Solubility:	*Very* slightly soluble in water. Soluble in alcohol and ether.
At 25°C and 760 mm Hg,	
1 ppm of vapor	0.00426 mg/liter
1 mg/liter of vapor:	235 ppm

III. Industrial Hygiene Practice

A. RECOGNITION: Used widely as a monomer for formation of polyester resins. Used with butadiene for the manufacture of Buna-S rubber (GRS or SBR rubber). The characteristic odor is readily identifiable at 25 ppm and is strong at 50 ppm.[5]

B. EVALUATION OF EXPOSURES: Can be determined by ultraviolet or infrared spectrophotometry. The nitration method of chemical analysis is useful for determination of small quantities. Physical methods utilizing thermoconductivity, vapor pressure, etc., could be used with suitable calibration.[6]

C. RECOMMENDED CONTROL PROCEDURES: Most processes and operations will require enclosure or process ventilation to maintain work room concentrations below objectionable levels. If unpleasant or irritating concentrations are encountered in short operations, this can be controlled by the use of personal respiratory protective equipment. Repeated skin contact should be avoided.

IV. Specific Procedures

A. FIRST AID: In case of eye contact, flush thoroughly with water for 15 minutes. Wash exposed skin surfaces thoroughly. Remove contaminated clothing promptly and wash skin thoroughly.

B. SPECIFIC MEDICAL PROCEDURES: No special medical procedures are necessary, although the usual preplacement and periodic examinations are desirable.

V. References

1. American Conference of Governmental Industrial Hygienists: *AMA Arch. Ind. Health 18:* 178 (1958).
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Ethylenediamine

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 10 parts of vapor per million parts of air, by volume (ppm).[1]

(1) *Basis for Recommendation:* Toxicological observations on animals.

B. SEVERITY OF HAZARDS:

(1) *Health:* Moderate for both acute and chronic exposures. The effects are primarily irritation of mucous

membranes and skin. Liquid contact can cause immediate skin damage with blistering. In some people cutaneous and respiratory symptoms of sensitization may follow exposures to low vapor concentrations.[2]

(2) *Fire:* Flash point 110°F (open cup).

C. SHORT EXPOSURE TOLERANCE: Human subjects found 100 ppm for 5–10 seconds inoffensive, 200 ppm caused tingling of the face and slight nasal irritation and 500 ppm caused intolerable irritation of the nasal mucosa.[3]

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Unknown.

II. Significant Properties

Chemical formula:	$H_2NCH_2CH_2NH_2$
Molecular weight:	60.10
Specific gravity:	0.8995 (20/20°C)
Boiling point:	116.9°C
Freezing point:	10.8°C
Vapor pressure:	10.9 mm of Hg at 20°C
Solubility:	Completely in water
At 25°C and 760 mm Hg:	
1 ppm of vapor	0.002458 mg/liter
1 mg/liter	408 ppm

III. Industrial Hygiene Practice

A. RECOGNITION: May be recognized by its ammoniacal odor and irritant effects. It is an extremely hygroscopic liquid which fumes in air. Ethylenediamine is used as a chemical intermediate in the preparation of dyes, inhibitors, fungicides, resins, and pharmaceuticals. It has direct use as a neutralizing agent.

B. EVALUATION OF EXPOSURES:

(1) *Direct Instrumentation:* None.

(2) *Chemical:* Collect in 0.05*N* HCl and titrate excess acid with 0.05*N* NaOH against methyl orange indicator.[3]

C. RECOMMENDED CONTROL PROCEDURES: Enclosure of processes accompanied by local or general ventilation. Eye and body protection should be worn if there is danger of splashing.

IV. Specific Procedures

A. FIRST AID: Clothing should be removed and contaminated skin thoroughly washed with water. In case of eye contact, immediately irrigate with water for 15 minutes and obtain medical attention.

B. SPECIFIC MEDICAL PROCEDURES: If allergic manifestations appear, complete withdrawal from exposures may be necessary. Skin or asthmatic difficulties should be carefully evaluated during placement examinations.

V. References

1. American Conference of Governmental Industiral Hygienists: *AMA Arch. Ind. Health 18:* 178 (1958).
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Because of space limitations, it is impossible to list all methods of exposure evaluation. The selections have been made on the basis of current usage, reliability, and applicability to the usual industrial type of exposure. Any specific evaluation and/or control problem will involve professional judgment. This can best be done by professional industrial hygiene personnel.

Respiratory protective devices are commercially available. Their use, however, should be confined to emergency or intermittent exposures and not relied upon as primary means of hazard control.

A relative scale is used for rating the severity of hazards: nil, low, moderate, high, and extra hazardous.

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News of Local Sections

Pacific Northwest Section

Officers for the coming year are Daniel J. Bessmer, President; Frank E. Adley, President-Elect; Darrel D. Douglas, Secretary-Treasurer; and John R. Horan and Ross N. Kusian, Directors.

Forty people registered for the two-day meeting, September 26 and 27, 1959. Papers read at the meeting were as follows:

"Evaluation of Using High Volume Sampling Rates for Air Containing Radioactive Particulates at Near Normal Background Concentrations," J. J. Sabo, J. E. Martin, and R. F. Grossman, USPHS Monitoring Evaluation Project, National Reactor Test Station, Idaho.

"A Sanitation Program for the Industrial Environment," John L. Johanson, Boeing Airplane Company, Seattle, Washington.

"Hazards of Fiberglas and Plastic Boat Manufacturing," P. Breysse, Environmental Research Laboratory, University of Washington, Seattle, Washington.

"A Beryllium Control Program," D. D. Douglas, Boeing Airplane Company, Seattle, Washington.

"Radiation in Commercial Aircraft," W. E. Morgan, Boeing Airplane Company, Seattle, Washington.

"Training of Health Physics Personnel," S. W. Farkas and J. E. Donnelly, NRF, Westinghouse Electric Corp., Idaho Falls, Idaho.

"The Atmosphere and Air Pollution," Dr. G. L. Hilst, General Electric Company, Richland, Washington.

"A Radiation Protection Program for Diagnostic X-Ray—Summary Report of the Oregon Survey," Dr. R. R. Sullivan, Oregon State Board of Health, Portland, Oregon.

A banquet was held at Berg's Chalet that evening. After dinner Lloyd B. Shone, Captain, M.C., USN, Director, Occupational Medicine and Dispensary Division, Navy Department, spoke on his recent trip to Helsinki, Finland, where he attended the Twelfth International Congress on Occupational Health. The J. M. DallaValle Award, presented for an outstanding contribution to industrial health, was given to Dr. Lloyd M. Farner for his pioneering of occupational health in the State of Washington. Dr. Farner was the director of the original industrial hygiene group in the Washington State Department of Health.

A business session was held in the morning on September 27th. In the afternoon a joint session was held with the Northwest Association of Occupational Medicine. Messrs. M. Chain Robbins and Walter H. Poppe joined with Doctors Rieke, Eckardt and Birmingham to present a panel discussion on toxic metals.

Tennessee Valley Section

The Tennessee Eastman Company, Kingsport, Tennessee was host for the annual meeting of the Tennessee Valley Section on October 9, 1959. A tour of the plant, one of the largest diversified manufacturing plants in Tennessee, was highly interesting. A complimentary luncheon was served in the Research Laboratories Cafeteria.

The meeting program included a business session and a number of excellent talks:

"The Occupational Health Program at Tennessee Eastman Company," J. E. Williams, M.D., Medical Director.

"Experimental and Clinical Evaluation of Effects of Chemicals on Skin," Part 1, "Prediction of Effects from Animal Studies," R. L. Roudabush, Ph.D., Laboratory of Industrial Medicine, Eastman Kodak Company, Rochester, N.Y. Part II, "Evaluation on Humans," D. W. Fassett, M.D., Director of Laboratory of Industrial Medicine, Eastman Kodak Company, Rochester, N.Y.

"Book Manufacturing," F. Stewart Crosley, Director of Public Relations, Kingsport Press, Kingsport, Tennessee.

"Recent Developments in Radioactive Waste Disposal." Speaker from Waste Disposal Research and Engineering Section, Health Physics Division, ARNL, Union Carbide Nuclear Company, Oak Ridge, Tennessee.

"Laboratory Techniques in Handling an Industrial Hygiene Urine Program in a Gaseous Diffusion Plant," T. Kwasnoski, Laboratory Service Section, Technical Division, Oak Ridge Gaseous Diffusion Plant, Union Carbide Nuclear Company, Oak Ridge, Tennessee.

Philadelphia Section

Twenty members turned out for the September meeting at the Engineers Club. Mr. Adrian L. Linch, Supervisor, Clinical and Industrial Hygiene Laboratory, Chambers Works, E. I. DuPont Company, presented an excellent talk on "Chemical Cyanosis: Cause, Effect and Prevention." This talk, clearly illustrated with slides,

was extremely informative and provoked numerous interesting questions.

The November meeting was held at the Philadelphia Naval Base and was an afternoon and evening meeting. A movie, "Function of Naval Shipyard," was followed by a tour through the machine shop, shipfitting and welding shop, the foundry, the Navy Yard, and the dispensary. After a social hour and dinner at the Shipyard Cafeteria a business session was held.

On December 4, 1959, the Tri-Section Meeting was held at the Stacy-Trent Hotel, Trenton, New Jersey. The New Jersey Section was our host for this meeting.

Ohio Valley Section

The fall meeting of this Section was held on October 6, 1959. Dinner was served in the cafeteria at the Robert A. Taft Sanitary Engineering Center. The group then moved to an auditorium where they heard Mr. Elmer P. Wheeler, national President of AIHA speak on the progress and plans of AIHA. He announced the results of balloting which favored the proposed program for certification of industrial hygienists. Also discussed were the statement of definition and scope of industrial hygiene recently adopted by the Board of Directors, and the plans for the AIHA meeting in Detroit in 1961. President Wheeler was given a thorough grilling by many interested questioners.

Chicago Section

Sixty-four members and guests attended the September meeting. Dr. Paul Whitaker, Medical Director of Allis-Chalmers and a past president of the Chicago Section spoke on "Occupational Health in a Diversified Industry." This proved to be an interesting and informative discussion of the industrial health problems encountered in his organization. The talk was illustrated by a fine selection of color slides portraying exposures and control measures.

Washington-Baltimore Section

The Section's annual half-day meeting was held on September 29, 1959 at the U.S. Army Environmental Health Laboratory, Army Chemical Center, Maryland. A tour of the Center and the Environmental Health Laboratory was followed by cocktails and dinner at the Officers Mess. Lt. Col. Maxwell Dauer spoke on "Radiation Aspects in Industrial Hygiene," and Capt. David W. Duttweiler discussed "Occupational Health Hazards Associated with Red Fuming Nitric Acid Disposal."

Michigan (Detroit) Section

The regular section meeting was held on September 22, 1959. Dr. Joseph H. McPherson, staff psychologist at Dow Chemical Company, spoke on "Psychological Aspects of Safety." This presentation evoked considerable interest and discussion from the members. The meeting was quite a success.

Rocky Mountain Section

New officers of the Section are: President-Elect, T. S. Chapman; Secretary-Treasurer, Morris J. Engelke; Director, Edward A. Putzier; and Local Sections Representative, Evan E. Campbell.

A two-day meeting was held October 16 and 17 in Albuquerque. Fifty-two members and guests attended for a fine program and good food. Sandia Corporation provided buses for a tour of Sandia Base. The tour featured a large array of physical test devices. Speakers and topics on the program were:

Elmer P. Wheeler, "New Chemicals and Petrochemicals." (Also informal remarks on national program of AIHA.)

Duncan Holaday, "American Standards for Uranium Mines and Concentrators."

Robert Elsbrock, "Sanitation and Some of Its Aspects in Industry."

Harold Rarrick, "Problems Associated with Testing Large Amounts of Irradiated Material."

John E. Smith, "Analysis of Chlorinated Hydrocarbons with the Gas Chromatograph."

Carl Jensen, "Stream Pollution by Wastes from Uranium Mills."

Jerome Dummer, "Health Physics Considerations of X-ray Installations."

New England Section

After the success of past meetings the Section elected to hold meetings for the remainder of the year at Publick House, Sturbridge, Massachusetts.

The speaker for the November 19th meeting was Joseph J. Fitzgerald, Assistant Professor of Physics, Harvard University. He is well known to industrial hygienists through his work at General Electric Company's Knowles Atomic Power Laboratory. Mr. Fitzgerald spoke on "Radiation and Air Sampling," and gave a most interesting insight into various phases of this currently "hot" subject.

Personnel Notes

Mr. John C. Cleeves, recently of Manchester, England, has become associated with Pangborn Corporation, Hagerstown, Maryland, as Devel-

opment Engineer, Dust Control Division. Mr. Cleeves, who was born and raised in Sussex, England, is a graduate of Cambridge University and has specialized in industrial dust and fume control problems with Tilghmans Ltd., Prat-Daniel Ltd., and Sturtevant Engineering Company, Ltd.

Obituary

Fred S. Mallette, an old and valued member of AIHA, died at his home on October 9, 1959. He had been in poor health for two years. Fred had been a member of AIHA since January 1940, and had served as Secretary (1951–1953) and as a Director (1954–1956). During his membership he served on numerous committees. Since 1952, he had been Executive Secretary of the Committee on Air Pollution Controls of the American Society of Mechanical Engineers. He was also editor of *Smog News*. For the past ten years he devoted his interest primarily to air pollution control and became a leading consultant in this field. His many fine contributions in both industrial hygiene and air pollution control have advanced these fields and will stand in his memory.

Aid to Medical Research

DOW CORNING CORPORATION has established a Center for Aid to Medical Research. In making the announcement, Dr. W. R. Collings, president, said: "During the past several years a number of remarkable advances have been made in medicine and surgery by using our silicones in a variety of forms. The interest among physicians and surgeons has grown so great that we have had to assign two of our best scientists to act in a liaison capacity between physicians and surgeons and our research laboratories. Our board of directors have recognized the responsibilities of the corporation by authorizing a non-profit unit separate from our commercial activities, but allied with our research department."

The purpose of the Center will be to aid medical research by supplying technical assistance, by acting as a clearing-house for information about the use of silicones in medicine and surgery, and by cooperating in research in organosilicon chemistry in relation to the human body. Offices of the new Center will be at Midland. The director will be Dr. Rob Roy McGregor, and the executive secretary, Mr. Silas A. Braley. There will be no charge for the services of the Center.

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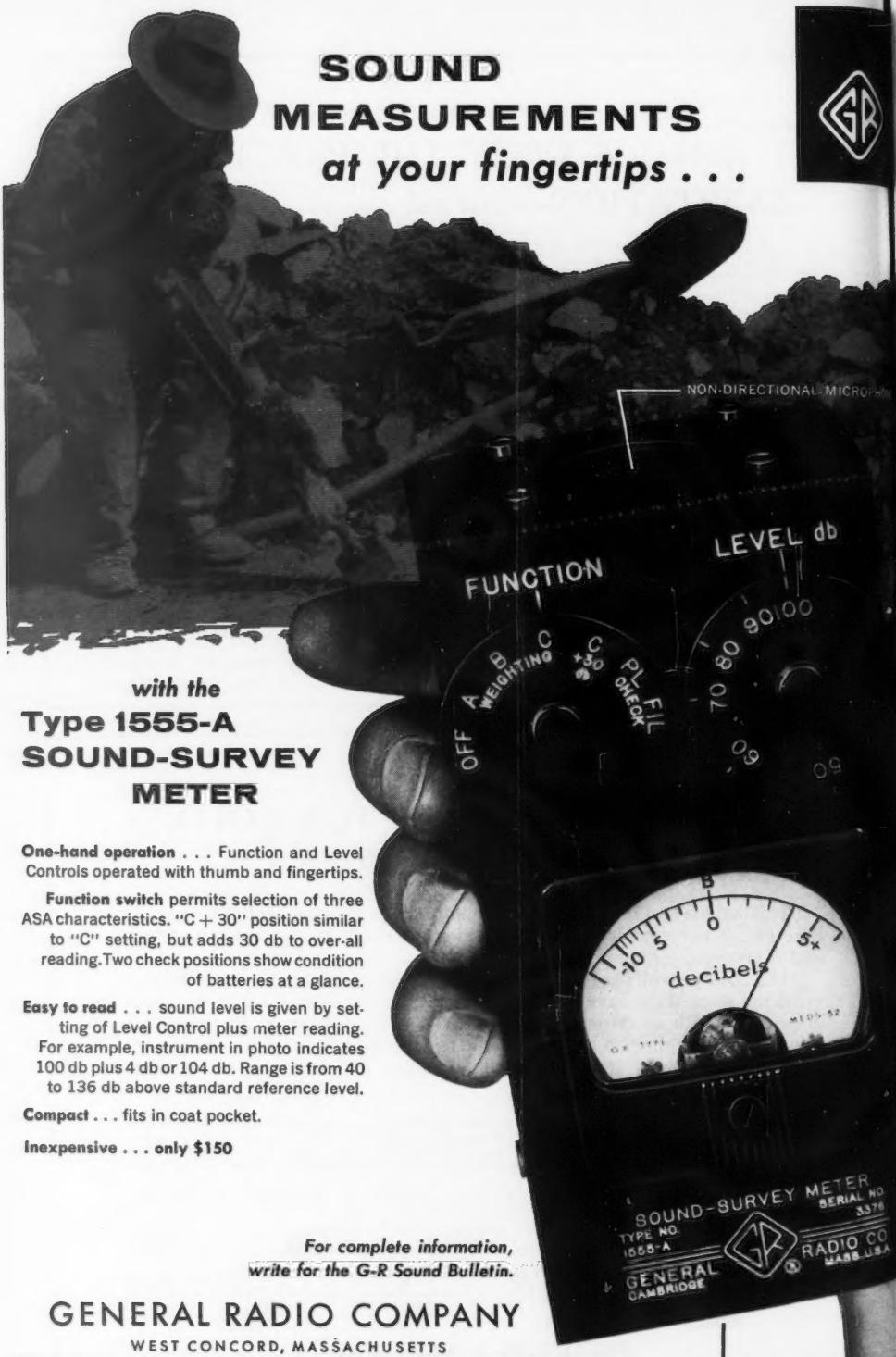

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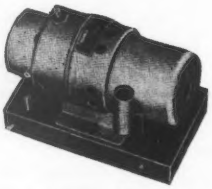

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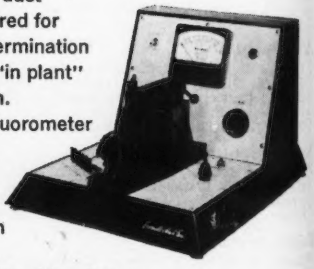

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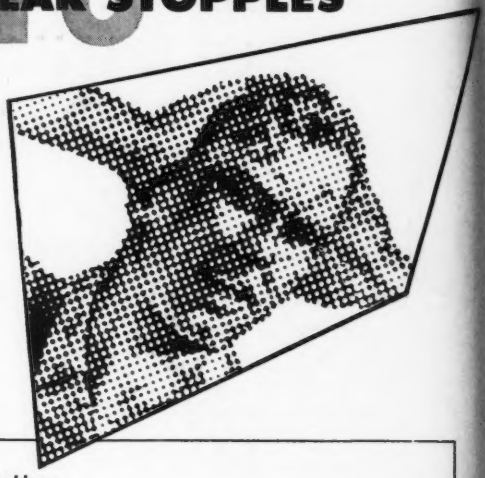

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